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"Plastics Powders-II"

MARCH 29, 1967 Sheraton Hotel Akron, Ohio

SOCIETY OF PLASTICS ENGINEERS, INC.

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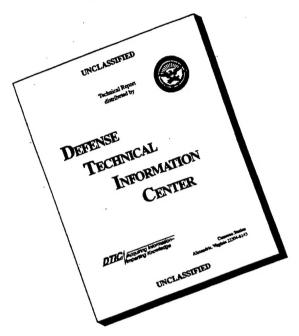
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Regional Technical Conference

Sponsored by

Akron Section

Society of Plastics Engineers, Inc.

March 29, 1967

# "PLASTICS POWDERS - II"

Regional Technical Conference of the Society of Plastics Engineers, Inc.

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AKRON SECTION

Akron, Ohio

March 29, 1967

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# TABLE OF CONTENTS

PAGE

	IACD
"Applications of Polyolefin Powders"	29583
Dr. R. Vernon Jones, Phillips Petroleum Co.	
"Thermoplastic Polyester Powder Coatings" W. T. Smith, Goodyear Tire and Rubber Co.	29584 11 46 42 4
"Coloring Plastics Powders"  John L. Satterfield, Americhem, Inc.	29585
"Performance Properties of PVC Bottles Produced From Powder Blends" Rodney P. Becker, Diamond Alkali Co.	212/13/1
"Electrostatic Powder Coatings" Emery P. Miller, Ransburg Electro-Coating Corp.	29587 26 96555
"Plastics Powder Coatings for Metals" Walter R. Pascoe, The Polymer Corp.	39 24346
"Rotational Molding of High Density Polyethylene Powders" Richard E. Duncan, US Industrial Chemicals Co.	50 92327
"Rotational Casting of Powders" Ronald E. Soderquist, McNeil Corp.	59 <del>927378</del>
"Powders for Rotational Molding Processes"  Robert Chandgie, Fusion Rubbermaid Corp.	66 7 7 9 1

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## APPLICATIONS OF POLYOLEFIN POWDERS

yr

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and

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### INTRODUCTION

Polyolefin plastics include polypropylene, low, medium and high density polyethylene and polyolefin copolymers and are the largest sales volume group of plastics materials in the United States today. The powdered polyolefins occupy a significant spot in these sales.

Powdered polyethylene sales have grown from very small beginnings in the late 1950s to over 30 million pounds in 1965 and all indications show that there was a substantial increase in 1966. This was started by the development of grinding mills that were inexpensive to operate and that produced a powdered product with a reasonable particle size distribution and shape. Previous to this polyethylene powders were made by precipitation from solution and were too expensive for use in large volume markets. Mechanical grinding is estimated to account for over 85% of the polyethylene powder produced, making it by far the most important method for producing polyolefin powders.

A limited amount of polypropylene and high density polyethylene reactor product is used in a powder form for dry blended compounds to be used in conventional extruders. These compounds are used for the addition of color pigment, stabilizers, fillers or fibers and are used this way because of lower cost or to reduce the heat history of the resin.

The bulk of the polyolefin powders available today are comprised of low, medium and high density polyethylene. Polypropylene and polyolefin copolymer powders, such as ethylene vinyl acetate, are at present still in the development stages. Since polyethylene powders comprise the bulk of the polyolefin powders used, we shall look primarily at these materials.

### POWDER MOLDING PROCESSES

There are several basic areas of fabrication which use powdered material. A brief description of each should help to place this portion of the plastics industry in its proper perspective. The first major area of powdered polyethylene use today is powder molding. It can be classified into three basic types - rotational

molding. Engel Process and Heisler Process.

At present, the most widely used powder molding process is rotational molding. The basic characteristics of this process are shown in Figure 1. In this process, the cold mold is filled with a powder equal to the part weight desired and is rotated about two axes at 90° to each other simultaneously. The mold is heated while rotating in an oven until the powder is fused and then is cooled by water spray or blown air while still rotating. The part is removed from the mold when cooling is complete.

Machines are available that use heating mediums such as circulated hot air, molten eutectic salt, gas flame or hot oil. These machines are also available with a number of different arrangements of rotating arms, ovens and cooling chambers.

The Engel Process (shown schematically in Figure 2), is a patented process sometimes called static molding since the mold is not rotated. The molding sequence is as follows.

The cold mold is completely filled with powder and then heated for a predetermined period to obtain the desired wall thickness, after which the unfused powder is removed. The mold with the partially fused powder on its walls is then reheated to smooth the inside of the part. After accomplishing this, the mold is cooled and the part removed. The production of large open end items is especially adaptable to this process. As shown in Figure 3, the Heisler Process differs from the others in that a preheated mold is filled with powder and the residual heat of the mold allowed to fuse the powder. In this case the mold is rotated about one axis through its longest dimension. The long axis is tipped to effect powder distribution and removal of excess powder.

Where thicker walls are desired, the mold must be reheated, more resin added and rotation continued until the desired wall thickness is obtained. To smooth the inside surface, a post-heating cycle is required. After the heating is complete, the mold is cooled and the part removed.

A second major powder use area is that of dry powder coatings such as are used for fabric coating, carpetbacking and the coating of other flexible substrates.

Carpetbacking with polyethylene powder consumes a large share of the powdered polyethylene produced. This technique is used on most of the carpeting in automobiles produced in the United States. It provides a moisture barrier and allows the carpets to be heat formed to fit the contours of the floor pan. This technique could also be used for household carpet but has not had acceptance in this area.

A schematic diagram of a carpetbacking operation is shown on Figure 4. In this technique, the uncoated carpet passes under an area where an even, continuous layer of powder is applied. It then passes under a series of infrared heaters where the powder is fused to the carpet. Finally, the carpet passes between a chill roll and nip roll for cooling before coiling.

The process is basically the same for fabric and other coatings except that the position of the chill roll and the use of a nip roll varies with the desired coating.

The third major area of powdered polyethylene use is that of metal coatings. A number of techniques are currently in use. One of these is the fluidized bed coating process shown in Figure 5 where after surface preparation, the part to be

coated is heated and then dipped into a bed of fine powder that has been agitated by air or inert gas passing through a porous plate in the bottom of the bed. A layer of powder sticks to the hot object. After removal from the bed, the part being coated is post-heated to smooth the surface.

The electrostatic spray method shown in Figure 6 differs in that the prepared part is not preheated but is sprayed with charged particles that cling to the part by electrostatic attraction. The part is then heated to fuse the powder into a continuous coating.

Flame spraying shown in Figure 7 consists of spraying the powder through the flame of a torch which partially melts the powder. The powder is then fused by the heat of the substrate which was heated previously by the open flame of the torch.

There are a number of other coating methods but they essentially differ only in the way that the powder is applied and the technique of heating.

# POLYETHYLENE POWDER PROPERTIES

In every fabricating process, the quality and success of any application depends upon selecting the right material and product design.

Polyethylene has certain properties that set it apart from other materials. Some advantages of powdered polyethylene are:

- 1. Excellent chemical resistance
- 2. Excellent toughness
- 3. High dielectric strength
- 4. High impact strength over a wide range of temperatures
- 5. Excellent low temperature properties
- 6. Light weight
- 7. Low cost
- 8. Abrasion resistant
- 9. Machinable
- 10. Weldable
- 11. Stain resistant
- 12. Colors are molded in
- 13. Most resins meet FDA requirements for food contact

# Some limitations are:

- 1. Limited outdoor formulations and colors available
- 2. Maximum softening temperature of 260°F
- 3. Relatively high coefficient of thermal expansion
- 4. Low resistance to creep
- 5. Environmental stress cracking resistance is normally low

Powdered polyethylene resins are classified by density, melt index and particle size. The following discussion will show the relationship of these parameters to the chemical, physical and powder properties.

All polyethylene resins, regardless of the process used to produce them, have their chemical and physical properties determined by three parameters. These parameters are density, molecular weight and molecular weight distribution. Most properties, however, are not entirely dependent upon any one parameter. Since some properties are more dependent upon the density than the other parameters, we can

make the generalization shown in Table I about the effects of a change in density on the chemical and physical properties of a polymer.

Low density polyethylene powder was the first to be used in large volume and has been used for applications from coating applications such as auto carpetbacking to molding applications such as hobby horses and even large industrial containers. As new resins were developed and more interest generated in the powder processes, medium and high density resins were introduced. Thus, today powdered polyethylene resins are available in a complete range of densities from 0.912 to 0.962. This entire range is usable in most of the powder processes if the melt index and mechanical properties of the powder grind itself are within a usable range.

The effects of molecular weight and molecular weight distribution on the chemical and physical properties are somewhat more difficult to assess due to their complex nature and their interaction. These parameters have little meaning when the user is looking at the range of powdered polyethylene available, therefore, the relationship of the properties with respect to melt index would be of greater interest. Broad statements and generalizations can be made about the effects of melt index on the chemical and physical properties that will hold true in most cases. Such a generalization is shown in Table II.

The polyethylene resins used in the powder processes are generally limited to those that will flow when melted in the absence of pressure to form a void free part. These resins generally range in melt index from as low as 3 to 5 melt index to as high as 70 melt index. The resins with the 3 melt index have better stress cracking resistance, impact strength, and resistance to creep than the high melt index resins but are also more difficult to mold. Parts with complicated, hard to fill areas would require a somewhat higher melt index for good moldability.

The powdered polyethylene resins in the 3-70 melt index range have much lower stress cracking resistance and long term load bearing properties than the very low melt index resins that can be used in blow molding, pipe extrusion and sheet extrusion. Thus, even though properly processed powder molded parts are almost stress free, care should be taken to eliminate areas of stress concentration in design. Because of the somewhat limited stress cracking resistance and load bearing resistance of the powder resins, parts fabricated from these resins may be limited to the severity of service that they can be subjected. The success of any part depends upon the design and processing in addition to the type of resin used, therefore, testing of the finished product under comparable conditions of use may be required to determine the best resin to use or if the application is too severe.

# RESIN SELECTION AND APPLICATIONS

The resin selection procedure can be broken down into four steps.

- Step 1 Consider the overall characteristics and properties available from the powdered polyethylene resins and determine if the part requirements could be satisfied by the use of this resin.
- Step 2 Determine the most important properties of the part. The requirements of these properties will, above all else, help determine the density and melt index of the resins that should be used.
- Step 3 Consider the particle size needed for the application as it may affect the moldability, resin cost and even heating time.

The choice of particle size may be a primary consideration for some powder processes and in others it is of less importance.

Step 4 - Consider the resin cost per finished part. If several resins are suitable, the one that had the lowest part cost would be selected. It is possible that a more expensive resin may have a lower part cost due to the need for less resin to obtain equal stiffness.

Using these steps as a guide the final resins selection may be a compromise in order to obtain the best combination of properties. For example, it may be necessary to sacrifice some stiffness for impact strength or stress cracking resistance or perhaps design changes might make possible the use of additional resins that would lower the part cost. With the variables involved and the compromises that many times must be made, we find that a given application may have several resins that will work equally well.

An illustration of some requirements and the recommended resin type or types are shown in Table III.

Some examples of powder molded polyethylene items being molded today are shown starting at Figure 8.

Creative designs such as are used for these items and many others, take advantage of the enormous number of sizes and shapes that are possible with the powder molding processes today. The future may see the use of polypropylene, polyolefin copolymers such as ethylene vinyl acetate, foamed polyolefins and improved polyethylenes that may open more possibilities for powder molding and coating applications.

### SUMMARY

The use of powdered resins for coating and molding has been rapidly increasing and its continued growth particularly in rotational molding, looks excellent. To-day there are many powdered polyethylene resins offering a variety of physical properties. The molder and coater should be aware of the properties of the available resins and should use this knowledge to help select the best resin for a given application. Proper resin selection, good part design, and proper processing coupled with applications properly selected for a powder process should produce items which give excellent service and have good customer acceptance. In the future, many present applications will find greater commercial markets and a host of new products will be developed by the creative designers that will utilize the unique abilities of the powder molding and coating processes.

# TABLE I

# EFFECT OF DENSITY ON RESIN PROPERTIES

Property	As Density Increases
Stiffness	Increases
Hardness	Increases
Tensile strength @ yield	Increases
Elongation	Decreases
Softening temperature	Increases
Low temperature impact	Decreases
Chemical resistance	Increases
Permeability	Decreases

# TABLE II

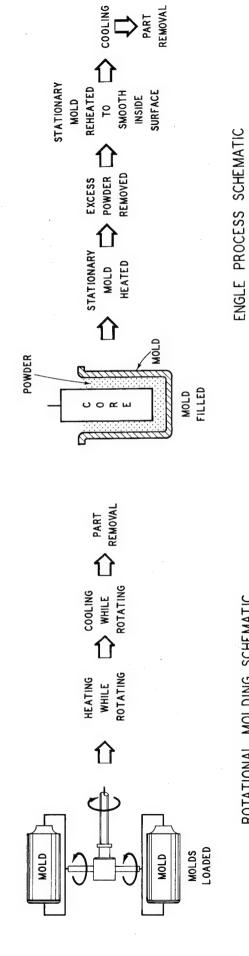
# EFFECT OF MELT INDEX ON PHYSICAL PROPERTIES

Property	Melt Index Decreasing
Melt viscosity	Increases
Tensile strength at rupture	Increases
Elongation at rupture	Increases
Resistance to creep	Increases
Impact strength	Increases
Resistance to low temperature brittleness	Increases
Stress cracking resistance	Increases

# TABLE III

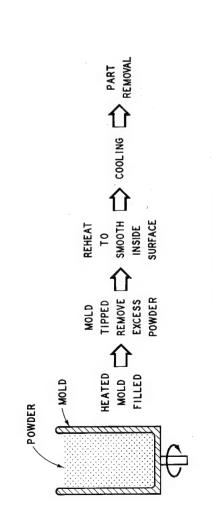
# EXAMPLES OF RESIN SELECTION

Important Properties	Recommended Density & Melt Index
High chemical resistance, lowest permeation, rigidity, impact strength, stress crack resistance.	High density⇒low melt index.
Flexibility, low cost, good moldability	Low density-medium to high melt index
Rigidity, high softening temperature, good moldability	High density-moderate to high melt index
Rigidity, high impact strength, stress resistance	Medium density-low melt index



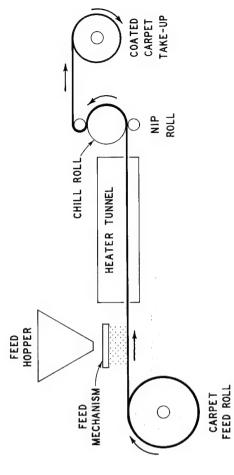
ROTATIONAL MOLDING SCHEMATIC

FIGURE 2

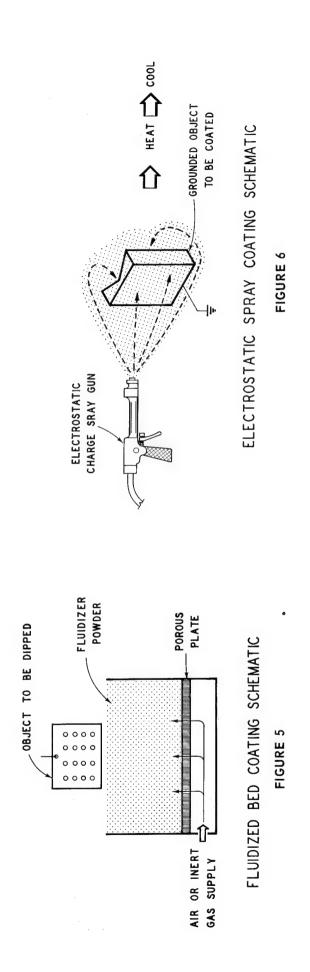


HEISLER PROCESS SCHEMATIC

FIGURE 3



CARPET BACKING SCHEMATIC FIGURE 4



GAS
FLAME POWDER

OBJECT

OBJECT

AIR
POWDER

FLAME SPRAY COATING SCHEMATIC FIGURE 7

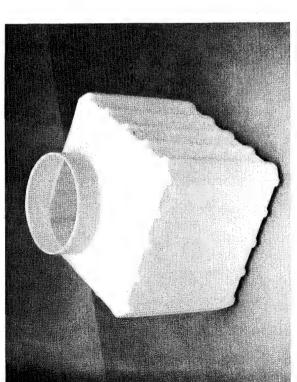


FIGURE 8: Bottled Water Dispenser Reservoir Molded by M-S Plastics of Ohio Co.



FIGURE 10: Light Duty Wheel

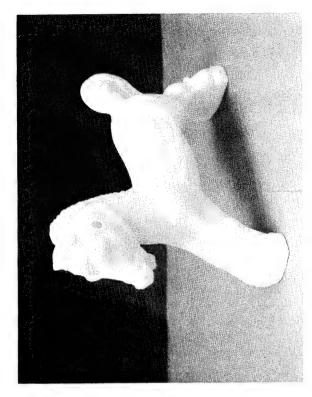


FIGURE 9: Toddlers Hobby Horse - Molded by Sun Rubber Co.

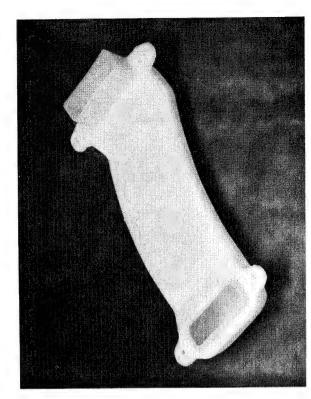


FIGURE 11: Small General Electric Refrigerator Air Duct Molded by Moll Tool and Plastics Corp.

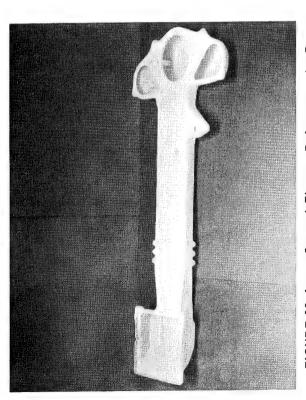


FIGURE 12: Large General Electric Refrigerator Air Duct Molded by Moll Tool and Plastics Corp.

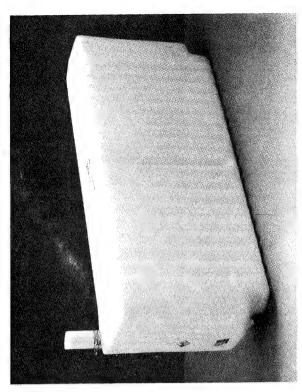


FIGURE 13: Camper Trailer Water Tank Molded by Inca Plastics

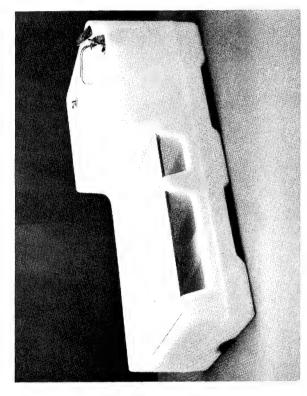


FIGURE 14: "Camp Sink" with Integral Water Reservoir Molded by Inca Plastics

# THERMOPLASTIC POLYESTER POWDER COATINGS



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The purpose of this paper is to present a comparison between the various chemical compositions of powdered resins currently being offered for application to heated substrates. It is apparent that these comparisons must be related to specific properties, and the relative importance of these properties as they bear on the end application desired.

It is not the intent of this presentation to cover the application of powdered coatings in any detail. It will, perhaps, suffice to present a general flow chart for the application of powdered coatings.

In this flow chart are the steps that are utilized in powdered applications and depending on the powder to be applied, one or more of these steps may be eliminated. The least number of steps that are required, however, are cleaning, heating, powder application and cooling.

It is obvious that any time it is possible to eliminate processing steps, the result is a more favorable set of economics. Consequently, the use of powders which do not require an adhesive or post curing or both have definite advantages over those that do. In addition, the straight economics of less processing steps and/or less material, as in the case of adhesive products obtained by simplified methods of application, result in a more uniform product with less quality control required and a higher percentage of first-grade merchandise produced. An example of this would be a statement made by the Federal Government three years ago in a report on powdered coatings which stated that "Resins which did not require an adhesive would make obsolete all those that do".

Before examining the comparison of the properties of the various powdered coatings, it perhaps would be well to discuss the important factors that users of this type of coating are looking for in their raw material supply.

## A. Adhesion

While it is obvious that the material must remain in place in order to perform satisfactorily through the useful life of the coated part, it is also worth noting that most applications also involve protection of the substrate against corrosion and that good corrosion protection begins with good adhesion. Adhesion may be obtained with the use of a primer, or it may be inherent in the particular powder selected. Were the choice available, most companies would prefer to eliminate the extra step for the

reasons already given.

# B. Dielectric Strength

Probably the second most important property is dielectric strength, since most applications are concerned with the degree of electrical resistance. This is particularly true in the field of motors, bus bars, transformers, pipe and underground installations.

# C. Handling

It is obvious that any coated object must be assembled, transported and installed. Consequently, the ability of the piece to go through these various steps without damage is important. This may be referred to as handle-ability. While handle-ability is a difficult property to measure, it is a combination of abrasion and impact; and consequently, materials that exhibit these properties to a high degree will give good performance when put through subsequent processes.

# D. Chemical Resistance

Chemical resistance is important, since this ties in very closely with corrosion resistance, salt spray resistance and film integrity when the coated object is subjected to various climatic conditions for sustained periods of time.

# E. <u>Uniformity</u>

Since many objects may go through a post-assembling operation where close tolerances are important, it is necessary that the coating give a reproducible thickness as well as color or gloss. It is obvious, of course, that the properties of the coating must be reproducible.

# F. Ability to be Repaired

Despite good handling characteristics and durability, heavy powdered coatings may be damaged either in the plant in which they are coated, in shipment or in installation at a customer's facility. To reduce scrap items and returned goods, it is necessary that simplified repair kits be available for reducing the cost to repair these items. A repair material should exhibit good adhesion to the metallic substrate as well as the coating itself and result in a smooth, not readily discernable repair.

There are, of course, other special properties which may be unique to specific applications, but the six listed here are generally considered among the most important, regardless of application.

The most widely used powders in order of volume are vinyl, epoxy, thermoplastic polyesters, CAB and nylon. There are, of course, a few small volume powders such as thermoset polyesters, fluorocarbons and copolymers of ethylene.

Within the general classification such as vinyl there are many subdivisions relating to specific polymer types and/or compounding, wherein the finished powder

is designed to meet the unique requirements of an individual application. To take vinyl as an example, this can be broken into two broad classifications - those powders which are dry blended and those which are froze ground. Then within each one of the internal classifications are the bariables of adhesion, stabilities, colorants and wetting agents, that may be adjusted with any given end application in mind. These various types of modifications are much more prevalent in the larger volume items, whereas those items which sell in smaller volumes such as thermoset polyester, nylon and fluorocarbons normally have only one offering and depend upon one or two specific properties in which they uniquely excel.

Understandably, where a large volume potential exists and is not completely satisfied with existing coatings, several of the powders may compete for the same market area. The largest current consumers of powders are pipe, motors, wire goods, transformers and communications. Many of these markets are subdivided, and a different polymer may be used consistent with the requirements of the coating on a specific piece. This is particularly true in the wire goods area where the applications may be dishwasher racks, coat hangers, chain link fence, garbage baskets, irrigation fencing and various building components, each having its own set of individual coating or processing requirements. Consequently, it may be advisable to use an epoxy, for example, in internal applications requiring high chemical resistance, a vinyl where economics are important and the adhesion obtained by encapsulation is sufficient for the purpose such as in the coat hanger trade, a thermoplastic polyester in the chain link fence area where corrosion and exterior durability are important, and CAB if the wire requirement is one needing the highest dielectric strengths available.

There are two markets in which powders are used wherein the requirements are pretty well standardized and yet within the capability of more than one powder currently being marketed. These are the pipe and transformer areas. To demonstrate how the typical properties normally run in testing any copolymer relate to its use in an end application, let us examine for a moment the characterization and properties of the three powders that are used in the pipe field versus the major competition. In this particular industry, this happens to be two products - the conventional coal tar applied from a hot mastic over a prime and pipe surface into which generally is imbedded felt and occasionally glass fabric and wrapped with a kraft paper for protection from weathering, and a polyethylene sleeve extruded over a mastic substrate. The specific polymers here, in order of their acceptance, are the thermoplastic polyesters, epoxies and vinyls.

Table I shows a general characterization of these types of coatings. Table II covers specific properties and represents a rundown of the type of test which would be applicable to the transformer industry. It is worthy of note, however, that really only the last test, that of salt crock disbonding, is specific to this industry. To carry this a step further, in Table III you see a rundown of the performance rating listing those particular properties which the gas and oil industries feel are important in a coating, and you will note that most of these particular properties relate directly to the fundamental properties of the polymer and that the coating performance can readily be forecasted from conventional test data. This we have found to be true in most applications, although there are certain small volume applications where one property is the dominating factor such as the previously mentioned electrical wiring where the major concern is for dielectric strengths and high temperature performance.

A pipe specimen is immersed in a salt solution and the coating is negatively charged. The potential may range from 1.5-6.0 volts, the salt concentration from .3 to 3% and the immersion time up to 200 days. The rating of the pipe depends upon the amount of coating which lifts during the immersion period.

It has been the purpose of this paper to present a comparison between the various types of powder coatings currently being offered in the market place. No attempt has been made to cover this broad, slowly evolving field in any depth. However, it has been pointed out that conventional testing consistent with standard ASTM practices, when properly interpreted, will give a sufficient base point to forecast performance of the product in the end application that the producer may have in mind.

TABLE I

COMPETITIVE STATUS OF POLYESTER IN THE COATED PIPE MARKET

Comparison of Coating Properties - General Characterization

	Coal Tar	Polyester	Epoxy	Polyethylene	<u>Vinyl</u>
Chemical family Type	Pitch Thermo- plastic	Polyester Thermo- plastic	Epoxy Thermoset	Polyethylene* Thermoplastic	Vinyl Thermo- plastic
Applied form Application method Primer Coating temp., OF Coating thickness, mils Service temp., OF	Liquid Flow Coat Yes 350-480 90 -20 to	Powder Spray No 550-575 10-12 -40 to	Powder Spray No 400-450 10 -70 to	Cubes Extrusion 10 mils* 350 25-60 -40 to	Powder Fluid Bed Yes 525-550 25 0 to 190
Pipe sizes	+160 3/4" - 36"	+160 3/4" - 12"	+250 3/4" - 12"	+150 3/4" = 10"	3/4" - 4"

<sup>\*</sup>A mastic 10 mils thick is applied and a polyethylene tubing is extruded over this.

TABLE II

# SPECIFIC PROPERTIES

	Coal Tar	Polyester	Epoxy	Polyethylene	Vinyl
Tensile strength, psi	-	7800 83	4 <b>-</b> 5000 82	3500 61	3900 45
Hardness, shore D Abrasion resistance,	-	0)	O2	01	
grams	•	0.006	0.051	0.07	0.042
Impact, Gardner, in-1bs.	-	20	20	•	10
Specific gravity	1.4-1.6	1.3	1.5	0.96	1.3
Water vapor transmis-			0 0		0.0
sion, $g/24 hrs/100 in^2$	-	3.6	8.0	0.01	8.0
Elongation, %	<b>ac</b>	2-4	None	100	25-30
Dielectric strength,		3.00	500 P00	1000	67.0
v/mil	-	100	500 <u>-</u> 800	1000 4-6# peel	510 Film
Bond strength	-	Film	Film strength	4-0# beet	strength
Adhesion	Good	strength Excellent	Excellent	None	Excellent
Chemical resistance	Good	Fair	Excellent	Excellent	Good
Water absorption, % in	dood	1411	DECOTIONS	DAGGIIGH	GOOG
7 days		.6	1.55	1.55	0.1575
Mandred bend	Fail	Pass	Fail		Pass
Salt-crock disbondingin.	•	5/16	6/16	8/16	8/16

TABLE III

# PERFORMANCE RATING

(5 to 1, Excellent to Poor)

		Coal Tar	Polyester	Epoxy	Polyethylene	<u>Vinyl</u>
1.	Field handling	2	5	3	3	5
2.	Driving	1	5	4	3	5
3.	Soil stress	1	5	5	3	4
4.	Bendability	1	4	2	3	5
5。	Bond strength	3	5	5	2	5
6.	Joint sealing	5	5	3	2	5
7.	Repair	4	4	4	4	4
8.	Toxicity	2	5	4	4	2
9.	Fume acceptance	2	5	4	4	2
10.	Service temp.	3	4	5	3	4
11.	Cathodic disbonding	4	4	5	4	4
12.	Chemical resistance	2	3	5	4	3
13.	Dielectric strength	5	4	3	4	3
14。	Process ease	5	44 damentosisto	4	4	3
	Total	40	63	56	47	50

COLORING PLASTICS POWDERS

dry

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### INTRODUCTION

While powder blend processing offers exciting cost savings to the plastics processor, several areas of potential trouble do exist, not the least of which is coloring. Careful consideration to coloring technique can result in savings of time, work and material, plus improved finished product quality.

Colorants for plastics come in many forms. For our purpose, these can be divided into four basic approaches:

1. Pure, dry pigment

2. Paste - pigment ground into a plasticizer

3. Chip concentrates - pigment fused into a compound which is chopped or diced into cubes

chopped or diced into cubes

4. Powder concentrates - pigment fused into a compound which is pulverized into a fine powder. (These are not to be confused with simple physical mixtures of pigments and resin.)

Each avenue of coloring has its own particular advantages and disadvantages which must be weighed in order to select the best approach for a particular process.

Good coloring is achieved by a complete distribution of the particles of pigment throughout the resin matrix. Some pigments are fairly easy to disperse with a minimum of work while others require considerable persuasion to separate the individual particles one from the other. Generally speaking, the inorganic pigments disperse much more readily than do the organic pigments, predominantly because of their larger particle size. However, colorants within each classification vary greatly and this must be accommodated for by the processor.

Best color control is achieved only when the color does not change in strength and shade on additional working. This can be measured easily on a two roll mill by milling the compound twice, cooling to room temperature between millings. Press polishing the two specimens from first and second milling together into one sheet gives them the same surface and is a very sensitive method of checking color difference. Any change in strength or shade of the color indicates poor color dispersion.

The end requirements for the part must govern the processor's thinking to a great extent. Obviously, inexpensive items that are salable if they are red, let us say, and not purple or orange can be colored by a different process than that used for those items that must pass rigid control standards. Good end product control demands complete control of each colorant used, eliminating as much as

possible any variation in shade or strength due to imperfect dispersion.

For the purpose of coloring, powder processes can be divided into two broad categories. First, there are those processes which exert very little mixing action on the fluxed stock and second, there are those processes in which the fluxed stock receives considerable mixing. The coloring methods which will be best for one process will not necessarily be best for the other.

## LOW SHEAR OPERATIONS

Fluidized bed coating, rotational casting and plunger injection molding are processes which do not exert a great deal of mixing action on the stock. For these operations it is very important that the pigment be completely distributed throughout the melted material prior to forming the part.

In many of these processes involving olefins, butyrates and polyesters, critical color requirements demand that the pigments be fused into the compound first, and then the material pulverized for use. This generally means buying precolored compound from the manufacturer. For some applications, dry blend pigments can be tumbled together until a thorough blend is obtained. This method is sometimes used for inexpensive items although it can result in incomplete color development and unreliable color control.

PVC powder blends for these low shear processes can be colored while compounding in a high intensity mixer. It is possible to get good dispersion of many dry pigments in this fashion; however, particularly hard to disperse colors can be used in paste form. The paste should be stirred into the plasticizer prior to introduction into the mixer. This will insure complete distribution of the color and will help prevent the resin from drying up the paste, forming hard agglomerated lumps of color which will reduce the available color strength.

Neither chip nor powder concentrates are suitable for this type of operation because there is not enough mixing to distribute the colorant after fluxing, and a salt-and-pepper effect occurs.

# HIGH SHEAR OPERATIONS

Extrusion, calendering or screw preplasticization molding gives the fluxed compound more mixing and thus allows the processor a wider selection of coloring routes.

Coloring PVC with pure dry pigment or paste in a high intensity mixer can provide a suitably colored compound for processing. This route is not without its drawbacks, however. Since this is a batchwise process involving small units, close control must be maintained to assure standard color. The pigments themselves are extremely dirty and the equipment requires prohibitive down-time for cleaning during color changes to eliminate contamination. Mixing of pastes into the plasticizer limits the processor's use of bulk systems and restricts automated weighing.

Coloring at a secondary step in the process sequence allows the processor to concentrate on dry\_blending uncolored compound at his most economical rate and is especially useful to one who makes short runs with frequent color changes. This is generally done in a ribbon blender or similar machine and predispersion of the pigment is an absolute must because there is not enough shear generated in this simple mixing operation to give full color development of raw pigment.

Chip or powder concentrates are the most suitable predispersions for this type of operation. Pastes, unless they are extended in plasticizer, may not mix readily and evenly into a powder. In addition to this, pastes are messy, difficult to handle, and are hard to clean from the equipment.

Properly formulated chip concentrates perform quite well if they are introduced as a prematched, one package colorant. Buty trying to produce a green, for example, from blue and yellow chips may lead to disaster in the form of streaks and variegated color due to heavy pigment concentrations in small areas of the mix. These prematched chips can be added to the powder in any fashion which results in a uniform blend. Ribbon blenders, drum tumblers, cement mixers and color proportioners can be used for this purpose. Chips have been the standard of the color dispersion industry for years. However, the processor using them must sacrifice inventory flexibility if he produces very many different colors.

Logically and practically, powder dispersions provide the answer to the problem of coloring powder compounds. These are really pigment concentrates in which the color pigment is fused into the resin binder and the resulting compound then pulverized into a powder, usually to the same approximate particle size as the vinyl resin. These are generally sold as dispersions of the single pigments and should be controlled as to strength and shade. Color matches can then be made using these as components. The best ones are designed to disperse completely under minimum shear conditions without developing additional strength or changing shade with further work. These dispersions, while not new in concept, are becoming increasingly more important in the coloring of plastics powders and improvements and refinements are still being made.

These powdered concentrates can be added individually to the compounded dry blend after mixing by using a ribbon blender, cement mixer, drum tumbler, color proportioner, or similar machine which will give good mixing action. The mix is quite clean and the equipment can be quickly cleaned to accommodate a rapid color change.

If desired, weighing errors can be minimized by preblending the powdered color concentrates. This can then be used to uniformly color a large quantity of compound. The premix can be used to good advantage in a color proportioner and lends itself to automated blending operations. An added advantage to this approach is that an aliquot sample of this color premix is truly representative, and color can be lab tested before mixing it with the dry blend. Excellent correlation is obtained between lab and factory results. Using this technique, one can, for example, set up a calender train using a continuous mixer fed with powder blend and metered premixed color concentrates changing color just as rapidly as the system can be purged.

One must be cautioned, however, that in using these concentrates he select those that are fully and completely dispersed and of a melt point suitable for his compound. More harm than good can come from attempting to incorporate a hard particle into a soft stock. This will produce lumps and streaks that no amount of working can eliminate. At best, poor dispersion can cause color variation during processing, and increased color development on reworking so that reprocessed material is off shade.

# SPECIAL EFFECTS

Special decorative effects such as metallics and pearlescents present peculiar problems. The pigments used are very fragile plate-like materials which can be

destroyed by high shear mixing. Aluminum, in powder form, also presents a severe fire hazard which can be avoided by using a powder dispersion. Dry pearlescent pitments present no such hazard and are available in powder form. They must be incorporated late in the mixing cycle to prevent their being broken up.

### COST

Cost is a prime consideration in the evaluation of any color system. All cost factors must be evaluated, however, and not just the price per pound of the colorant. Obvious coloring costs include color matching, standardization and control, weigh-up, cleanup, waste, lost capacity due to off-color runs, and any predispersion work that is done. To these obvious costs must be added factors which concern uniformity, plant cleanliness, inventory control and convenience. Each individual plant is also limited by its color match facilities and the training and experience of the staff. Each processor must apply his own formula to these criteria to make a meaningful decision.

## CONCLUSION

In summary then, we can say that pure color pigments or paste dispersions can be used to color plastics powders and indeed are necessary in applications in which little mixing of the stock is done during fabrication. However, there is a price to pay in plant and equipment cleanliness, and care must be taken to minimize scrap from non-uniform color development.

Powdered color concentrates are being used more and more by dry-blend processors. They can help reduce cleanup time, colored stock inventory and off-shade scrap due to poor color control. Expensive, high intensity mixers can be used to best advantage repairing natural compound with little or no cleaning between stocks while color can easily and accurately be added later using relatively inexpensive equipment which is quickly cleaned between colors. This allows the processor to take full advantage of bulk handling systems and automated weighing devices.

In choosing a colorant and coloring method, the processor must consider these points - type of plastics being used, the part being fabricated, equipment available, and the preciseness of color control desired. The coloring method must also be one which is compatible with his laboratory facilities and the training and experience of his staff.

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# PERFORMANCE PROPERTIES OF PVC BOTTLES PRODUCED FROM POWDER BLENDS

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### INTRODUCTION

Previous publications, 1 together with a noticeable market penetration, indicate that rigid PVC bottles can be successfully blow molded from powder blends. However, in order to determine possible end uses for polyvinyl chloride bottles, certain performance properties must be completely evaluated.

As with any new plastics application, failure to conduct performance tests can lead to misapplications and a poor market image for the material. The marginal outdoor weathering characteristics of early PVC corrugated sheeting should serve as a vivid example of insufficient testing. Certainly no one in the vinyl industry would like to see PVC containers fail from such a technical standpoint. Thus, this paper gives consideration to maximum filling temperatures, use of regrind, effects of bottle geometry, compression strength, permeability and drop impact performance of vinyl bottles.

## REGRIND

The economic success of a blow molding operation, regardless of the thermoplastic involved, is dependent upon the use of a "closed loop" system. Scrap, in the form of tails, trim and imperfect molded parts, will always be accumulated during production. It then becomes absolutely essential to continuously remold this scrap with virgin compound to obtain economical use of the material. Continuous addition of the generated scrap defines a "closed loop" system of operation.

There are certain precautions that the fabricator must take when applying a "closed loop" system to rigid PVC. Any form of scrap (i.e., tails or unblown parisons) must be cooled to prevent degradation and subsequent contamination of a large quantity of regrind. In addition, the tendency of processed vinyl to collect dust and dirt dictates the need for isolating regrind from a dusty atmosphere. Neither of the above characteristics present serious difficulty if reasonable care is exercised.

Several logical questions to be asked about the use of PVC regrind are:

1. Is there a substantial color difference between bottles produced from virgin material and those made with 30% regrind?

- 2. How does the use of regrind affect the physical properties (i.e., drop impact) of the finished bottle?
- 3. Is the clarity of a PVC bottle inversely affected by using regrind?

The answers to these questions are of paramount importance to the bottle producer.

Since rigid vinyl is a heat sensitive material, compounds can and will change color during processing. However, the use of powder blends effectively minimizes these color "drifts" because the virgin material has virtually no prior heat history. There is a slight color difference (well within commercial standards) between virgin material and bottles containing 30% regrind, but the use of a "closed loop" system implies that all bottles will contain equivalent quantities of scrap. Thus, the color of finished bottles will be identical.

Development of ultimate physical properties from a rigid PVC dry blend is dependent upon sufficient frictional heat input during extrusion. Regrind material is subjected to the same frictional heat during the extrusion process. Therefore, in identical processing, bottles containing 30% regrind are equivalent in clarity and drop impact characteristics to bottles produced from 100% virgin material. Our laboratory results, as well as production tests, have verified this identical clarity—drop impact performance.

# FILLING TEMPERATURES

There are numerous packaging applications that require the product to be hot-filled. If vinyl is to be considered for these items, the effect of filling temperature on bottle distortion must be determined. Two compounds, one a food grade for-mulation, were blown into bottles at our laboratory and subsequently tested for distortion at various filling temperatures.

Bottles were filled with liquid (either water or oil) at various temperatures and allowed to cool to ambient temperature. Physical changes in bottle geometry, as observed by visual inspection, were then reported. Results of this investigation are reported in Table I. These results can be summarized as follows:

- 1. No significant difference was detected between the use of oil or water as the filling media.
- 2. Both oval and round bottles were tested and significantly higher filling temperatures were obtained with the round bottles.
- 3. Non-food grade, 22 gram, 8 oz. Boston round bottles can withstand up to 180°F filling temperatures.
- 4. Food grade compounds generally require fill temperatures 20°F lower than non-food grade compounds.
- 5. Regardless of the shape or compound, all bottles tested could pass a 150°F fill temperature.

As previously reported, 2 the size and shape of a bottle influence the maximum filling temperature. Therefore, actual filling conditions should be simulated prior to designing a PVC container for a specific product.

# COMPRESSION STRENGTH

The load bearing properties of rigid vinyl bottles were studied using the approved SPI Column Crush test procedure. This test consists of placing uncapped bottles in an upright position between the platens of a load measuring unit. Holes in the top platen allow venting during the compression which is carried out at a crosshead speed of one inch per minute. The load which causes the first observable deformation of the specimen is the compressive yield load.

PVC and polyethylene bottles of equivalent size, shape and weight were tested. When compared directly to PE (Table II), vinyl exhibits its superior mechanical strength by supporting a 1.9 times higher loading (88 versus 46 pounds). This results in a greater margin of safety in filling, capping and stacking operations. Also, PVC's high mechanical strength would permit the use of lighter weight containers capable of withstanding loads equal to the heavier PE containers.

## PERMEABILITY\_COMPATIBILITY

A primary consideration when evaluating plastics containers is the ability of the bottle to retain its contents over long periods of time without undue weight loss of contents or effect on bottle properties. Many proprietary formulas, together with basic classes of chemicals, have been tested in rigid PVC and results are reported in the literature. However, to maintain quality of packaging, each new product should be subjected to permeability and compatibility studies prior to marketing.

The typical method to measure weight loss of contents is to place the product into the desired bottle and make weighings at periodic intervals. Ideally, test conditions should encompass room temperature as well as some elevated temperature. Our laboratory has chosen 120°F as the elevated test condition because a storage warehouse may reach this temperature during the summer months, and there is an approximate correlation between eight weeks at 120°F and 52 weeks at 73°F. In general, the results at 120°F for eight weeks are approximately equal or slightly greater than those obtained at 73°F for one year (Table III). Thus, the use of 120°F provides a more immediate guideline for predicting the suitability of PVC for a product.

It is essential that some temperature above 100°F be used as a test condition because there have been isolated instances of product incompatibility in this temperature range. The incompatibility has resulted in the PVC bottle turning cloudy after a very short storage time (one week) at about 100°F. The simple elevated weight loss test can, therefore, save costly misuse of rigid PVC containers since it also functions as a final compatibility check.

# BOTTLE GEOMETRY

As indicated earlier, the geometry of a vinyl container can affect such things as maximum filling temperature and compression strength. Geometry can also have a drastic influence on the drop impact characteristics of a finished bottle. Extremely small radii (less than 3/16") at the base of a container can lower the mean failure height (measure of drop performance) by as much as 50%. This becomes more exaggerated as the size of the container is increased and should certainly be considered for anything larger than a 15 oz. container.

Some evidence of side wall collapse has been reported for specific products

stored in cylindrical PVC bottles. Such collapsing appears to be associated with the lack of a flat panel in the container wall since an oval type design does not exhibit this phenomena. Furthermore, vinyl is not alone in this type of failure because similar problems are noted with polyethylene. Testing of the product in the proposed container design can effectively eliminate these deficiencies.

# DROP IMPACT

With the myriad of compounds (both powder and pellets) available to PVC bottle manufacturers, it becomes virtually impossible to do anything but generalize about drop impact characteristics. This area of impact is the most confused and poorly defined segment of vinyl bottle marketing. Compounds which range from crystal clarity (low impact) to extremely high impact (slightly poorer clarity) are all being sold and have definite places in the market. Certainly there is no need for the producer of small containers (less than ten ounces) to sacrifice any clarity at the expense of unneeded impact strength. However, larger bottles require compounds with a high degree of impact strength. Consequently, the need for better definition of market requirements and establishment of impact standards (i.e., something like plastics pipe standards) is readily apparent.

When subjecting bottles to a drop impact test, results can be affected by:

- 1. Totat capacity (bottle size)
- 2. Level of fill
- 3. Height of drop
- 4. Test temperature
- 5. Point of incidence of the bottle
- 6. Weight (or wall thickness) of the bottle
- 7. Bottle geometry
- 8. Composition of the vinyl compound

Bottle failure generally consists of a ductile break which occurs as either a pinhole or short slit in the side wall. Some shattering is encountered with the high clarity materials, particularly at low temperatures.

## CONCLUSION

The information presented above is offered as a guideline for determining the applicability of PVC in the bottle industry. As indicated, rigid vinyl has numerous advantages over other polymers currently in use and should obtain a substantial segment of the market.

A significant point for consideration is that no product should be marketed in a rigid PVC container (or any plastics bottle) until sufficient testing has been conducted. Absence of such elementary information as compatibility and permeability data could result in large weight losses or cloudy bottles upon storage. An improperly designed bottle could exhibit poor drop performance, side wall collapse, or distortion upon filling. Furthermore, the bottle producer should exercise reasonable care with the use of regrind if a successful operation is to be maintained.

The potential problem areas referred to in this text should not be confined exclusively to the application of rigid PVC containers. In effect, they apply to the use of any plastics material which is to be considered for packaging. One may still hear reference to plastics (in general) being referred to as "cheap". Proper testing and marketing programs can greatly assist the industry in upgrading plastics in the consumer's mind.

# ACKNOWLEDGMENTS

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TABLE I

EFFECT OF FILL TEMPERATURE ON BOTTLE GEOMETRY

		Fill	Tempera	ture -	Degree	s Fahrer	heit
		140	150_	<u>160</u>	<u>170</u>	180	<u>190</u>
I.	15 oz. Oval Bottle						
	A. Non-food compound						
	1. Oil 2. Water	NC NC	NC NC	NC NC	NC NC	SC BD	BD BD
	B. Food Grade Compound						
	1. Oil 2. Water	NC NC	NC NC	SC SC	BD BD		- - -
II.	8 oz. Boston Round Bottle						. 13
	A. Non-food compound						
	1. Oil 2. Water	NC NC	NC NC	NC NC	NC NC	NC NC	SC*
	B. Food grade compound						
	1. Oil 2. Water	NC NC	NC NC	NC NC	SC*		

# Specifications:

15 oz. Bottles: weight 32 grams, average wall thickness 16 mils 8 oz. Bottles: weight 22 grams, average wall thickness 18 mils

NC = No Change

SC = Slight Change

BD = Bottle Distorted

<sup>\*</sup>Slight bulding at base of bottle

TABLE II

# COMPRESSIVE YIELD LOAD

Bottle Size and Shape	Material	Bottle Weight, grams	Yield Load, lbs.
15 oz. detergent	High density poly- ethylene	34	46
15 oz. detergent	Rigid PVC - non-food	34	88
8 oz. Boston round	Rigid PVC - non-food	22	111
8 oz. Boston round	Rigid PVC - food-grade	22	107
40 oz. Imperial quart (round)	Rigid PVC - non-food	51	60

# TABLE III

Product	8 weeks at 120°F	52 weeks at 73°F
Oven cleaner	3.00	1.65
Furniture polish	NC	NC
Hand lotion	1.75	1.80
Oil soap	2.05	1.75
Car wax	1.80	1.90
Hair dressing	2.85	1.80
Mouth wash	2.40	2,20
Hair shampoo	2.05	1.90
After shave lotion	4.80	4.00
Spray starch	2.85	1.50

NC - No change Bottle - 15 oz. oval, weight 32 grams, average wall thickness 16 mils

## ELECTROSTATIC POWDER COATINGS

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# INTRODUCTION

Recent advances in industrial technology in all areas has developed uses for all types of materials in powdered form and it, therefore, has become desirable to apply these powders to surfaces under various conditions and for many different purposes. Moving hand in hand with this desire has been the development of production methods whereby new and different materials have become available in powdered form. In addition, those powders which have been available are now produced more economically in large quantities having a more carefully controlled particle size distribution. Metallic powders of the most exotic elements and compounds are now produced and used under controlled reaction conditions in the manufacture of all sorts of metallurgical products. Organic and inorganic powders are used in the fields of pharmaceuticals, food production, and construction materials manufacture.

All of this activity has stimulated an interest in the development of new methods of handling and applying materials in powdered form. Perhaps one of the most active interests has arisen because of the desire to use these materials to form upon the surfaces of articles of manufacture, films or deposits which are simultaneously protective, decorative, and functional. We propose to discuss such applications and in particular the use of plastics powders for this purpose.

# HISTORY

In any coating application it is normally desired to produce on the surface a deposit of some solid material. It can be in the form of a continuous film or discontinuous deposits depending upon the end use desired. It is common practice, in order to facilitate application, to place the solids into a liquid solution or suspension which is then handled as a liquid from container to deposition on the surface. The liquid distributed over the surface then flows out and levels. The solvent then completely or partially evaporates leaving the distributed solid coating. The final film is formed by application of heat. Such techniques are legend and have been for many years.

Since the solvent is usually a fast evaporating liquid material serving only the function of a carrier vehicle, it adds cost and oft times additional hazard to the operation. Industry has long sought to partially or completely eliminate the use of solvents from such coating processes. Coatings having high solids have been developed so that less solvent will be required for effective application. Water base materials have been developed and used to avoid the fire hazard incident to the use of other solvents. Higher solids coatings may be applied if elevated

temperatures are used to reduce their viscosity at the time of application.

Much has been done to develop plastics coatings for solvent free application. A promising solution first appeared with the development of the fluidized bed technique by Edwin Gemmer of Knapsack-Griesheim in 1953. This process is now well known. With this process the solid material to be applied is reduced to powder form. The powder is placed in a special container and aerated from below so as to form an air supported expanded "bed" which physically behaves like a body of liquid. The part to be coated is preheated to a point above the melting point of the powder and immersed into the bed. The powder in contact with the part, fuses, attaches itself to the part and remains as a coating when the part is withdrawn. The amount of powder adhering to the part is a function of the time of immersion and the thermal capacity of the part. Quite heavy coats can be produced. This method is being used to coat a wide variety of parts ranging from wire goods to electrical components.

As this process was improved and applied, industry became increasingly aware of the advantages of solventless application. New powders were developed aimed specifically at meeting the demands of this technique. At the present time vinyls, cellulosics, epoxies, nylon, polyethylene and chlorinated polyethers are being applied by this technique.

In the early 1940s, electrostatic methods of applying liquids were introduced. The individual liquid paint particles were given an electrical charge opposite to the article to be coated and, therefore, were attracted to the article reducing material losses due to over-spray. These same electrostatic techniques have now been applied to the application of powders of all types and in particular to the application of plastics powders.

# ELECTROSTATIC POWDER DEPOSITION

It is a well known electrical phenomena that a body carrying a negative electrical charge will be attracted to an adjacent body bearing a positive charge. This phenomena is utilized in powder deposition by intentionally placing a charge on the powder particles and an opposite charge on the object to be coated. When the charged particles are brought near the article an attraction will result and the powder particle will deposit on the article surface. The design of electrostatic deposition equipment for powders must solve the problems of giving the powder the maximum possible electrical charge of the correct sign and of conveying the powder to the vicinity of the surface to be coated.

# HAND POWDER GUNS

These features have been designed into electrostatic powder guns which can be manipulated to distribute the charged powder about the object to be coated. Typical of such devices is the powder gun shown with its controls, its voltage supply and its powder source in Figure 1.

In this device the powder to be applied is placed in a container shown at the lower right of the picture. This container is arranged with a porous bottom so that the powder can be adequately aerated and formed into an expanded bed, much as in the fluidized bed process referred to previously. Adjustment of the air flowing to the bed is carefully controlled to insure uniform bulking of the powder volume. Vibration of the powder container is sometimes provided to produce constant conditions within the bed.

In the bed and well below the surface of the bulked powder there is located an air venturi type pump. This pump is connected on its inlet side to a regulated air supply and on its outlet side to a flexible smooth-walled hose running to the gun. The venturi chamber of the pump has an opening into the powder bed. When air is supplied to the venturi inlet jet, powder is drawn into the pump and exhausted into the supply hose. This air carries the suspended powder to the gun which in Figure 1 is shown on top of the high voltage transformer near the center of the picture. A control panel which houses regulators and gauges for the various air supplies as well as the electrical controls is at the top of the picture.

The gun in close up view is shown in Figure 2. It consists of two parallel tubes of insulating material extending forwardly from a grounded metal handle. A hollow, tubular shaft is mounted for rotation inside of the upper tube. This inner hollow shaft is coupled to an air motor in the handle and carries at its other forward end the powder diffuser. This diffuser is a plastics bell shaped member which has a conductive coating of high electrical resistance applied to its outer surface from hub to forward sharpened edge. Its central opening is directly connected to the bore of the shaft. Powder from the supply source is pumped to this hollow shaft and thence to the rotating diffuser.

A high voltage cable runs from the voltage supply to the rear of the gun. It extends through the lower tube of the gun being insulated so it cannot be contacted. At the forward end of the high voltage tube a flexible brush connects the high voltage to the conductive coating of the diffuser.

In operation of the device the air to the fluid bed is turned on so as to fluidize the powder. The air to the gun motor is turned on causing the diffuser to rotate at approximately six hundred revolutions per minute. Pulling the trigger on the gun then activates a relay which energizes the voltage supply primary and applies a high voltage of about eighty thousand volts to the diffuser surface. The same trigger action energizes an air solenoid which causes air to flow through the venturi pump and bring the air born powder to the gun from the supply bed. This powder travels through the rotating support tube to the center of the diffuser. A special button at the center of the diffuser deflects the powder outwardly over the diffuser surface and past its outer sharpened edge. The presence of the voltage on the sharp edge of the diffuser produces charged air ions in this region. These air ions bombard the powder particles as they pass putting on them an electrical charge.

When the gun is held with its diffuser adjacent to an object to be coated and this object is grounded, an electric field is established between the diffuser edge and the object. Under the action of this field the charged powder is attracted to all surfaces of the object.

This action is apparent by referring to Figures 3 and 4. Figure 3 shows the powder leaving the gun under the action of the carrying air forces alone. The absence of marked forward velocity of the powder particles is evident in this photo. Figure 4 shows the action which follows the application of the voltage to the diffuser. The strong attraction of the charged powder to the grounded objects is evident. The application efficiency is quite high. As the particles follow the electrical lines of force of the field, they wrap onto all portions of the object surface. Coating efficiencies as high as 80% are not uncommon on larger surfaces. Efficiency is very sensitive to the forward velocity imparted to the particles by the carrier air stream and it is, therefore, desirable to keep this as low as possible if highest efficiencies are to be obtained.

The nature of the diffuser will determine the shape of the powder cloud patern. More restrictive cones will give narrower patterns. By shaping the deflector

button at the center of the head the overall shape of the pattern can also be varied. Rotation of the charging diffuser is not always necessary. By using straight slotted nozzles with internal baffles a fan shaped spray for special application can be obtained. Increasing the air pressure on the venturi inlet will increase the amount of powder delivered to the gun. Increasing the voltage on the gun will increase the effective distance over which the powder can be deposited.

Since most plastics powders can be flammable when dispersed, the spray gun is constructed so that no high energy electrical discharges will occur from it when it is brought close to the grounded article. The nature of the diffuser as well as inherent impedance in the gun itself limits the available energy within reasonable limits.

### PARTICLE CHARGING AND DEPOSITION - COLD SUBSTRATE

Whereas the general theory of particle attraction by electrostatic forces is extremely simple and easily understood, many complexities arise when the technique is brought to practicality. The force with which a particle is urged toward an oppositely charged surface is a direct function of the magnitude of the charge carried by the particle and of the strength of the field existing in the deposition region.

Theoretically both of these quantities increase as the voltage applied to the gun increases. However, this voltage cannot be increased indefinitely. Some unusual undesirable electrical effects arise at higher voltages and the voltage must be held within limits dictated by possible hazards. It is customary to use voltages in the range of 70 to 90 kilovolts.

In order to charge the particles the gun must be provided with some element capable of producing air ionization when raised to the applied voltage. This usually takes the form of a sharpened conducting point or edge at the front of the gun. The material particles are carried past this element and through the cloud of charged air particles. They are bombarded by the charged air particles and thus receive their charge. The charge limit is reached when the accumulated charge is sufficient to repel approaching charged particles. Theory shows that this limiting charge is directly proportional to the strength of the field at the point of charging and to the square of the radius of the particle.

The electrical properties of the material have an influence on the behavior of the process. Usually those materials perform best which have a rather small electrical conductivity, low specific gravity and high dielectric constant. For best results the particles should be of uniform size and reasonably spherical and have a diameter in the range of 20 to 120 microns.

Once the particle is charged it moves toward the article under the influence of the combined force of the electrical field and its own initial momentum.

The strength of the field will be dependent upon the voltage on the gun and the spacing of the gun from the object. Since these devices are usually used with their forward end at about 10" from the work, the average deposition field will be about 6,000 to 7,000 volts per inch. If the electrical forces are strong enough, the particles will be deposited on the article surface.

The phenomena of deposition is again not as simple and straight forward as the basic theory would indicate. Various characteristics of the material are again effective in this portion of the process.

In the case of electrostatic application of liquids, the deposited particle or droplet is self-adhesive and once deposited it is automatically retained upon the surface. The liquid droplet, due to its liquid character, will also have reasonable electrical conductivity. It will release all of its electrical charge to the grounded conducting target surface forming a coating on the target. The charge flows off to the ground and thus maintains the surface being coated at ground potential.

Matters are somewhat different in the case of solid powder particles. The powder materials generally have little adhesive character and may vary from insulators to good conductors. Whereas the electrical resistance of the powder may not have marked effect on the charge acquired by the particle, it certainly will influence its behavior upon deposition.

A solid particle which is charged will be deposited upon the surface. If the particle is a good conductor, the charge, regardless of its location on the surface of the particle, will at once flow off to ground. If the surface is at room temperature and dry, only mechanical forces will then be present to adhere the particle to the part. Subsequently arriving particles will be attracted without electrical interference to the surface and equally well to the previously deposited material. The mechanical forces adhering the material to the surface will very quickly be equaled by the weight of the deposited material and the layer will fall off.

If, however, the particle is a good insulator, the particle deposited on a surface at room temperature will lose only that portion of its charge which is on the surface of the particle in contact with the metal part. The remaining charge on the particle will be attracted to the surface and will thus serve to electrostatically hold the particle in place. Other arriving particles will be deposited on uncovered areas and will adhere in a similar manner.

Since under these conditions the deposited powder has retained some of its charge, the point will be reached when an approaching particle will find a surface charged to a reasonable magnitude and to the same sign as itself. Further deposition will stop under these conditions and the approaching particles will be repelled from the surface to some other object.

It can be seen that the amount of powder which can be accumulated and retained on a cold surface, therefore, becomes a function of the rate at which the deposited material loses its charge. The bulk resistance of the powder material and the surface electrical resistance of the particles, therefore, have an influence. Because the charge on the material can be lost to ground through the atmosphere the relative humidity of the deposition area also has an influence on the amount of powder collected. Since such deposits are generally not retained as powders but are subjected to heat after deposition to bring about fusion of the powder on the surface, the film thickness of the films which can be formed thus are also subject to bulk and surface resistance of the material and to atmospheric conditions of the deposition area.

The value of the bulk resistance of a powder material can be altered by adjusting the powder composition. The surface resistance of the particle can be changed by controlling the treatment which the particle receives. The humidity and temperature conditions of the atmosphere in the coating area can likewise be controlled. It is, therefore, possible to select powders and control conditions of applications in a manner which will allow a definite film to be obtained. Some powders have been prepared specifically for this purpose and have given excellent coatings. For any particular application a complete study of these conditions is needed before a full evaluation of the possibilities of application to a cold substrate can be made.

It will be evident that powder application to cold substrates generally results in films of fairly uniform thickness because of the self-limiting character of the accumulation. A particle which is repelled from one point on the surface will tend to seek out a portion of the surface where it will be attracted.

Since the temperature of the part will alter the temperature of the powder, heating can be used to influence the powder conductivity. As a matter of fact, by heating the surface to be coated to a temperature only slightly above ambient temperature the layer of powder which can be made to adhere to the surface can oft times be increased by several magnitudes.

Since the forces holding the powder in place on the part are electrostatic and subject to change, it is necessary to take into account the handling which the part will receive between deposition and fusion. A result may be satisfactory in one set of conditions and not in another where the handling to which the part is subject is more rough.

Because the control of the amount of material that can be retained upon a cold surface is sensitive to the previously mentioned variables, the deposition of powders on cold substrates has found only limited application.

### PREHEATING

Where it is possible to do so a great deal of the uncertainty of film formation on the article surface can be overcome by preheating the article in advance of deposition to a temperature above the fusion point of the powder. The charged particle is attracted to the heated surface just as well as to a cold substrate. By heating the surface the arriving particle fuses upon contact with the surface and adheres. Because it is molten, its conductivity increases and all charge leaks off to ground. It not only does not present a repelling charged surface to subsequently arriving particles but provides a suitable surface to which additional particles may adhere. The collected powder will continue to build so long as the operation is continued and so long as there is sufficient residual heat in the part to cause adhesion of the arriving particles. The film thickness deposited with heated substrate is under the control of the operator and dependent upon powder characteristics. Films of thirty to forty mils can be readily applied with some materials. Because of the inherent character of powder application, it is difficult to obtain films which are less than two or three mils thick. The thinner films are more often easier to obtain by application on cold substrate.

### HAND GUN APPLICATIONS

Many coatings are now being applied by powder hand gun techniques. Electrical machinery components such as transformer covers and tanks are being coated with vinyl materials. Large motor and generator rotors are epoxy coated to a thickness which is adequate to provide the needed electrical insulation and mechanical rigidity. Cellulose acetate butyrate is being applied to metal laboratory furniture. A unique application involves the use of teflon powder to coat screen type paper mill rolls. Numerous special applications are being done in cases where liquid application is not successful or where film characteristics unique to this method are desired. Other exotic powders such as brazing fluxes and mold lubricants are being applied with the hand gun to fill a special need.

### AUTOMATIC APPLICATION EQUIPMENT

Whereas the previous discussion referred to the hand held powder application equipment, these same principles can be applied to automatic equipment. By suitably designing the guns and positioning them about the articles to be coated as the article is carried past, effective systems can be produced. One such system is being used for the application of plastics coatings to the exterior of iron pipe. A general view of an apparatus of this type is shown in Figure 5.

The coater proper is a cylindrical enclosure arranged to have the pipe pass through it along its axis. The pipe is mechanically cleaned and heated to the proper temperature, about 450°F, and then carried to the coater. In the coater eight powder spray guns are arranged spirally about the pipe. They extend through the wall of the coater and can be seen in the figure as white tubular members. Each gun has a high voltage lead brought to its rear end. This passes through the gun to contact and charge its diffusing head. These leads in turn connect to the high voltage supply shown in the upper rear of the picture. A voltage of approximately ninety thousand volts is applied to each diffuser. A powder delivery tube also enters the rear of the gun. This tube is connected to one of the eight venturi pumps in the powder bed supply at the lower right of the photo. In this case, the diffusers on the guns are not rotated and pattern is controlled principally by diffuser shape and gun position with respect to the pipe.

The air delivery to the venturi pumps, the voltage and other elements of the operation are all controlled from the single console which can be seen at the position of the operator.

With this arrangement each length of pipe as it is delivered to the coater is attached to the preceding length by a temporary coupling. The pipe essentially passes lengthwise through the coater as a continuous length. Powder pumped from the bed is sprayed by the guns; charged by the voltage and attracted to the hot surface. Upon deposition, the powder fuses and flows to a smooth coating. The pipe is then quenched in a water shower to solidify the coating so the pipe can be handled.

### COATING CONDITIONS

Because the powder particles are not as small as liquid paint particles the deposition efficiencies of powder systems are not as large as that for the corresponding liquid systems. Despite this fact, such powder systems are quite efficient overall since all material which escapes deposition can be reclaimed. Suitable air exhaust systems and cyclone filters are attached to these systems for this purpose. Figure 6 is a look into the end of such a coater during operation. The powder emitted from the gun can be seen to be attracted to the surface of the heated pipe to produce the glossy plastics finish. The manner in which the sprayed particles follow the electric field to the work is visible in the spray from the first gun.

A number of such units are in operation at the present time. Pipe two inches in diameter is being coated with an epoxy coating twelve mils thick at the rate of seventy-five linear feet per minute. Larger diameter pipes up to eight inches are coated at correspondingly lower speeds.

Arrangements are being made to use similar distributing devices to plastics coat the exterior of pipes up to thirty—six inches in diameter. In this case, it is contemplated that the guns with their auxiliary equipment will be moved along

the hot pipe surface as the pipe is rotated.

### ELECTROSTATIC FLUIDIZED BEDS

Recently there has been introduced to the market an arrangement which combines the normal fluidized bed with the characteristics of electrostatic charging elements. In this device, as shown diagramatically in Figure 7, a group of pointed electrodes are introduced through the porous bottom of the fluid bed and distributed over its surface. These electrodes are connected to a high voltage source which holds them at an electric potential of about one hundred kilovolts above ground.

When the bed is aerated by introducing air from below, the powder is bulked so as to cover the tips of the electrodes. The high voltage is then applied to points. The high field at these points charges the powder particles in the bed and repels them upward into the space above the bed. If the surface of any grounded object is placed in the space above the bed, these charged particles are attracted to it and collect upon it.

This device, unlike the normal fluidized bed, can be used to apply powder to objects or surfaces which are at room temperature. The amount of powder accumulated will be subject to the same conditions mentioned above in connection with the use of the powder hand gun. The adhesion of the powder layer to the cold substrate and the control of the several variables to produce consistent results are a problem. Because the system is a closed one, little powder is lost from the process.

Beds of this type are being used to coat sheet stock after the manner shown in Figure 7. They have also found use in various modifications of this same idea in the coating of continuous cable as well as discrete objects carried adjacent to the bed on a grounded conveyor.

### MATERIALS AND APPLICATIONS

Many materials can be applied by these methods. Particularly in the plastics coating field many powder types with varying characteristics have been developed and are commercially available.

The most widely used to date are the epoxy materials. These thermoplastic powders are applied with automatic equipment as pipe coatings, as coatings for wire goods and utensils and in the electrical field where they serve as good insulators. They have good self-adhesion and give durable, hard finishes.

Polyvinyl chloride plastics have possibly the widest application range, although they also have severe limitations. They have good toughness, excellent corrosion resistance but their poor adhesion demands the use of a liquid primer for best results. They are used on consumer products, particularly wire goods and for a wide range of industrial applications. These materials are being applied without a primer with the powder gun where strippable coatings are desired.

Polyethylene powders have found ready application. Since the material does not readily dissolve in most solvents, the powder method has really opened a new avenue for this very versatile product. High density materials have recently of fered new and very interesting possibilities. Although adhesion is normally rather poor, newer formulations have vastly improved this characteristic. This material

finds its widest use in the food processing and packaging field.

Nylon powders are available. Their quality is excellent but their price is high. Nevertheless, these materials can be used as coatings on surfaces where good protection and a slick self-lubricating type of surface is desired.

Other powders which are available and which will find increased use as the techniques develop are the polyesters, chlorinated polyethers (Penton by Hercules Powder Co.), and fluorocarbons (Teflon by duPont).

Other plastics such as the polyurethane, polystyrene, melamine, acrylic and polypropylene are, of course, known and would have highly desirable properties, but as yet only little attempt has been made to work out electrostatic coating techniques.

The ideal powder is one which will exhibit in film form the properties desired and in addition will adhere without using a primer. The powder application method demands that the material have good handling characteristics in the powder form and be relatively independent of ambient conditions. The behavior of the material as it changes from the powder to the liquid state upon being heated is also important. Those materials having a rather wide melting range will behave best.

Most powders present some explosion hazard. This feature must be considered in the design of the application equipment. High energy electrical discharges and static build—up must be avoided. There is also some danger of toxic fumes being released when the powder is fused. Despite these features, good powder applications are possible when the equipment and installation are carefully and judiciously designed. Coatings which are functional as well as decorative are effectively and economically applied by these methods.

Recent developments have resulted in these techniques being used for the application of porcelain enamel powders to both cold and hot substrates. Thin films of porcelain enamel coatings have been applied to sheet material at Battelle Institute using electrostatic fields to precipitate the powder from a circulated carrying air stream as the air stream flows over the sheet surface. Subsequent heating fuses and flows the powder to form the film. Guns of the type described above have been used to apply heavier one-coat porcelain films to heated cast iron substrate. Here the substrate is heated to approximately 1500°F, and the powdered frit is distributed about the surface by the hand gun. The electric charge attracts the frit to the heated surface where it sinters and adheres. Subsequent heating produces the necessary flow and leveling.

### DUSTERS

Plastics powders are not the only materials which can be applied by these methods. Wherever a dusting or powder coating application is desired some modification of this general technique will be applicable. Equipment arranged to apply talc or mica flake to rubber sheet is shown in Figure 8. Here the rubber sheet passes through the unit supported on suitable rolls. Above and below the sheet there is located one of the automatic powder spray guns described above. In this application the guns are oscillated to more uniformly distribute the material over the sheet. The diffusers of the guns are charged by being connected to a voltage supply. The powder to be applied is aerated in a self-contained bed to the bottom of the unit and pumped to the guns in the manner described above in connection with the pipe coater. Excess powder is returned to the bed. Exit and entrance openings in the coater are provided with an exhaust curtain to collect material that

might escape from these openings.

Another powder applicator for wider sheet is shown in Figure 9. This unit applies powder to only the top surface of the sheet. The application guns are located above and oscillate to insure increased uniformity of application. The supply bed and collector shown withdrawn from the duster in this photo, is a unit arranged below the sheet being coated. It is made readily removable so that several such units containing various materials can be used alternately if desired. When being used the bed and collector is rolled into place beneath the spray guns. Such a unit would be used as well to apply plastics powders to rubber webs, cloth strip and paper and metal foil.

### CONCLUSION

Electrostatic coating equipment for the application of powders is available in various forms. Hand held units are being used in numerous industrial applications. With these guns a charged powder is distributed about the articles to be coated and is attracted to the article surface. Automatic equipment for powder coating surfaces of articles which are reasonably consistent in shape and requirements are finding use in the pipe coating field as well as in other areas of industry. A broad spectrum of plastics powders are now in production and available for a variety of uses. A great deal of effort is now being expended by resin producers toward modifying their products specifically for electrostatic use. Many potential uses outside the field of finishes application add breadth to the potentials for this method. The successes thus far experienced certainly add impetus to new efforts in this direction and go far toward predicting an expanding future for this technique.

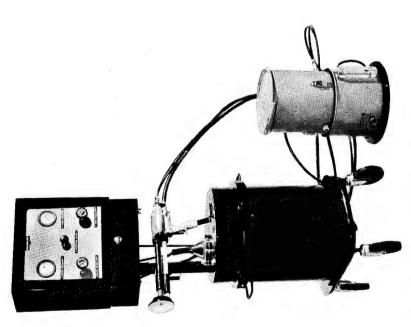


FIGURE 1: Electrostatic Hand Powder Gun with Voltage Supply, Controls and Powder Supply



FIGURE 2: Electrostatic Hand Powder Gun

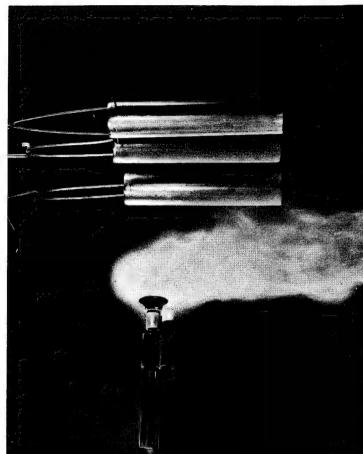


FIGURE 3: Electrostatic Powder Gun in Coating Posi-tion without Voltage Applied

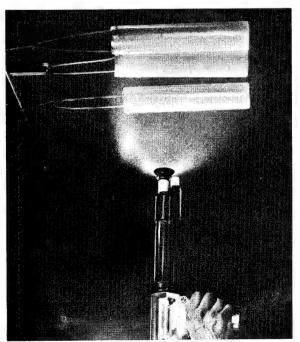


FIGURE 4: Electrostatic Powder Gun in Coating Position with Voltage Applied

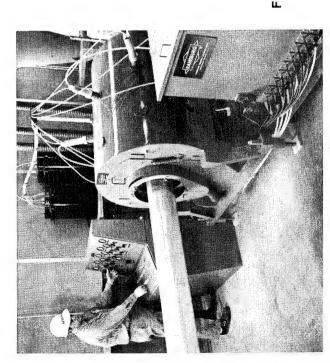


FIGURE 6: Internal View of Electrostatic Powder Pipe Coater

FIGURE 5: Electrostatic Powder Pipe Coater

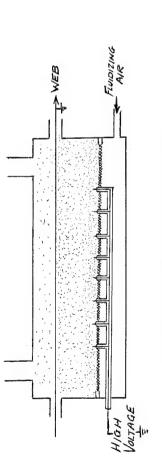


FIGURE 7: Electrostatic Fluidized Bed

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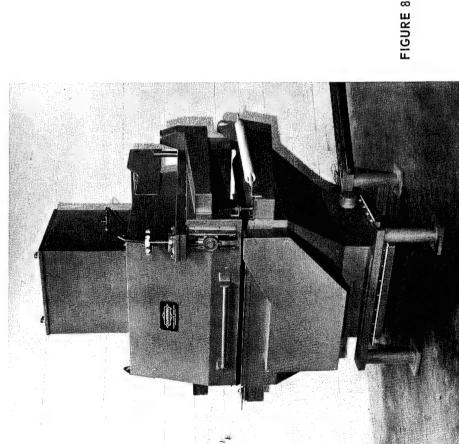


FIGURE 9: Electrostatic Sheet Duster



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### PLASTICS POWDER COATINGS FOR METALS

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Several unique and versatile powder coating processes for cladding metals with plastics, including electrostatic spray and fluidized bed, have developed from laboratory curiosities in barely 11 years to routine production operations today in almost 200 major companies. It is apparent that these durable fusion coatings fill a need at costs compatible with market requirements. While numbers are one index to acceptance, perhaps a more significant measurement is found in the wide range of applications.

From a marketing standpoint the present powder sales volume has often been exaggerated (by a factor of 5 or 10). Dreams of future potential are confused with present realities. While the powder market is slowly increasing, it has become quite competitive.

The rate of growth of the vinyl coating powder market has not kept pace with the vinyl resin industry. Present total United States sales of vinyl compound for powder coating are less than two million pounds per year divided among 15 or more suppliers compared to vinyl resin industry sales of greater than two billion pounds per year.

We do see many potential opportunities in powder coating. However, to achieve these potentials it is necessary to consider and to work with many variables. Selling a new powder customer may require five years. It is necessary to get material qualification and acceptance both from the powder customer and from his coated product customers as well. Sample parts are coated and evaluated. Coating cycles are developed and coating parameters are defined. Each coating equipment system is a custom effort. Considerable engineering effort and assistance is required on a no-charge basis to aid the customer in design and start-up of the coating line. Even then substantial continued technical service is needed.

New powder suppliers, facing these formidable investments, in time and in money, often solicit business from existing customers rather than assisting in the expansion and in the development of the total market. The only advantage they can readily offer is lower prices. They underestimate the substantial servicing cost which equals the cost of the vinyl resin itself.

This pattern has delayed expansion of the powder coating industry. There has been considerable interest in the new technology of the process, but the reduction of this interest to powder sales has been slow. This partly is due to erroneous concepts about coated product advantages and the applied cost. As an example, people frequently evaluate powder coating as a replacement for paint which it is not.

### WHY ARE POWDER COATINGS BEING USED?

Fusion bond finishes are used to clad metal parts for two primary reasons:

- 1. Product improvement including the ability to achieve superior performance, properties or characteristics which could not be obtained previously, i.e., higher operating temperature by applying epoxy coatings to motor laminations or improved corrosion, dielectric and weathering properties on vinyl coated transformer assemblies.
- 2. Cost reduction due to lower labor costs, reduced production time, lower materials cost, lower capital investment, economies of eliminating finishing operations or allowing the use of less expensive substrates.

### WHERE ARE POWDER COATINGS BEING USED?

The advantages of fusion bond finishes have gained wide commercial acceptance in five major areas of use:

- 1. Outdoor Weathering "where maintenance means money", such as metal furniture, outdoor fencing, transformer parts and marine hardware.
- 2. Low Order Corrosion such as direct burial items, i.e., underground gas distribution pipe, and parts exposed to chemical fumes or seacoast atmospheres.
- 3. High Order Corrosion plating racks, pumps, valves, pipe and fittings used in the chemical industry.
- 4. Electrical Insulation new "integral" insulation on rotors and stators, busbars, coils and pole-line hardware.
- 5. Mass Produced Items such as appliance and automotive parts where unique cost advantages are important.

### WHAT APPLICATION TECHNIQUES ARE USED?

There are several different plastics powder coating techniques. These varied methods include fluidized bed, electrostatic spray, flock coating, cloud chamber and powder flow coating. In addition these plastics powders are also used in retational molding and casting. We will confine our consideration to the two most popular powder coating techniques - fluidized bed and electrostatic spray.

The first of these two techniques to be commercially introduced was the fluidized bed or Whirlclad(R) coating system. While the principle of fluidization of solid particles was known and used for some time, the concept of using an aerated bed to apply finally divided plastics to preheated parts originated in Germany in 1953.

Polymer Processes, Inc., a subsidiary of the Polymer Corporation, introduced the Whirlclad coating system to the United States in late 1953. 31 patents have  $\overline{(R)}$ Trademark of The Polymer Corporation

been granted relating to this process by the U. S. Patent Office, more than 20 are pending. Over 500 companies are licensed to employ this coating system by Polymer. This process is now used in every major industrial country in the world for applying many different plastics to a wide variety of parts, both by manufacturers and by custom coaters.

Fluidized bed coating is based on dipping a preheated object into a bed of "finely divided", dry fluidized powders which melt and fuse on the heated surface to form a continuous uniform film.

The parts to be coated are preheated in an oven above the melting point of the powder to be applied. The preheat temperature depends upon the type of plastics used, the thickness of coatings to be applied, and the shape, mass and substrate of the part.

The hot parts are then immersed with suitable motion into the fluidized bed. A rising current of air passing through a porous plate at the bottom of the specially designed tank fluidizes the plastics powder. In this condition, the powder looks and acts like a liquid. If the tank is tipped or rocked, the level of the powder moves and changes as a liquid would.

As the powder particles contact the heated part, they fuse and adhere to the surface. After removal from the fluidized bed, the coated parts are often briefly postheated to completely fuse the coating and to obtain the best surface appearance. If a thermosetting compound such as an epoxy is used, this postheat cures the resin.

### ELECTROSTATIC SPRAY COATING

The electrostatic application of plastics powders is rapidly becoming an important technique in producing fusion coatings. This process is being used to apply powder coatings to such items as pipe, transformer covers, large coil springs, appliance parts and household utensils. Possibly the major use is the coating of pipe for various applications.

The process works on the principle that oppositely charged particles attract each other. Powder is fed from a reservoir, often a fluidized bed, to a gun by air pressure, where a high voltage, low amperage electrostatic charge is applied. The powder picks up the charge from a transfer of electrons from the gun to the powder. This transfer takes place both through contact with the highly charged gun nozzle and through the surrounding ionized air. The part to be coated must be electrically conductive and must be grounded. As the charged powder approaches the grounded part, it is attracted to the part where it clings until fused into a plastics coating.

An important characteristic of this process is the ability to produce coatings without the use of preheat as required in the fluidized bed technique. The quality of such coatings depends greatly on the type of plastics powder used. Certain powders loose their charge very slowly (epoxies, cellulosics, nylons) after being deposited on a part. The deposited layer then repels incoming powder and results in a limited thickness (about 5 to 10 mils maximum). Heavier coatings require the use of preheat. Other powders (melt mixed vinyl in particular) have the proper combination of electrical properties to permit the rapid build-up of heavy layers of powder on cold substrates. Still other powders lose their charge too rapidly and fall off the part before the coating can be thermally fused. Non-homogenous powders such as dry blends apply less uniformly as the discreet particles of resin,

plasticizer, pigment and stabilizer selectively take different electrostatic charges. Also dry blend application is much more affected by high humidity than are the melt moxed vinyls. Of course, the powder deposited on a cold substrate must subsequently be heated sufficiently to cause it to fuse into a coating.

### WHAT ARE THE ADVANTAGES OF THESE PROCESSES?

Because of the unique methods of application, these processes offer many different advantages. Advantages common to both techniques are:

- Since dry plastics powder is used and only becomes molten briefly during the coating process, excellent uniformity of coating is characteristic even over sharp edges. Fusion bond finishes, properly applied, are unmarred by sags, drip marks, bridging or surface irregularities. These processes are excellent for coverage of sharp corners, edges and projections on many metal stampings, fabricated wire goods and machined parts.
- 2. Solvent resistant plastics can be used which previously could not be considered as coatings. For example, nylon, polyethylene, chlorinated polyethers and fluorocarbons can now be applied.
- 3. Less expensive substrates can be used. A high grade steel alloy part can be replaced with inexpensive castings protected with a fusion bond finish.
- 4. These coatings can be machined. Manufacturers coating parts such as pumps, and valves with chlorinated polyethers for chemical resistance machine the coatings, particularly on the valve seat, to close tolerances.
- 5. Minimum surface preparation is required. For example, hot alkaline degreasing, to remove grease and oil from the surface, followed by a water rinse is often sufficient.
- 6. The processes are adaptable to automation and to automatic processing of parts in large volumes because cycle times are rapid, involve few operations, and have minimum labor requirements.

  These processes can also be used with batch equipment for coating small quantities of parts or for processing large and heavy parts.
- 7. With the same equipment installation, a wide variety of colors and materials can be applied to parts of many different sizes and shapes. Also, colors or resins can be interchanged rapidly.
- 8. Practical repair techniques are available for these coatings.
- 9. Relatively short coating cycles are possible since multiple coats, bakes and handling are eliminated. Immediately upon cooling, the parts can be packaged, shipped or used. No additional drying or baking time is needed. This is important where large objects or large volumes of parts are being processed.
- 10. Production reject rates are low.
- 11. Savings are substantial because of the complete use of coating

powders. No solvents are lost through evaporation, plasticizers are not lost in quantity during baking, and coating materials are not lost in clean-up. Also, these plastics powders have a virtually indefinite shelf life.

### SPECIFIC FLUIDIZED BED ADVANTAGES

- With the fluidized bed, heavier coatings can be applied. It is possible to apply a controlled, uniform thickness of coating from 5 to 50 mils without the use of solvents in a single dip operation. It is not necessary to apply multiple coats with multiple handling, baking and drying time to obtain a suitable film thickness as with many other coating techniques.
- 2. In addition to providing an attractive, durable finish, the thickness of plastics coating applied by a single operation provides outstanding sound, electrical and thermal insulation.
- 3. The time needed for coating application is much less with the fluid bed technique.
- 4. Surface appearance is smoother with less orange peel.
- 5. Complicated shapes can be uniformly coated due to the liquid qualities of the fluid bed of dry powder.
- 6. The thickness of the fusion bond finish often eliminates costly machining required with conventional thin coatings. Smooth finishes can be obtained on rough surfaces without extensive substrate preparation. The coatings are applied to inexpensive rough sand castings with virtually no surface preparation.
- 7. The process often allows desirable product design changes. Motors have been redesigned to smaller, more compact units with improved copper to iron ratio through elimination of bulky slot liners. Because of uniform edge coverage, new products of perforated or expanded metal have been introduced.

### ELECTROSTATIC SPRAY ADVANTAGES

Some of the advantages of electrostatic spray coating process are:

- 1. The ability to produce a coating without the use of preheat.
  Where preheating is required, it is usually less than with fluidized bed coating.
- 2. Masking is simplified on cold parts. Inexpensive, low temperature tape or even cardboard can be used, and then removed prior to post-heating. Alternatively, the powder can simply be brushed off the areas where no coating is desired.
- 3. Large thin sections (sheet metal) which are difficult to coat via fluid bed because of the rapid loss of heat, can be sprayed electrostatically and then postheated to fuse the coating.

- 4. One side only coating is possible on some parts, without masking, if a little overspray is permissible on the rear side.
- 5. Another advantage worth considering is that a large inventory of powder is not required to begin coating (such as is needed to charge a fluid bed tank).
- 6. Likewise, coating can continue until the powder supply is nearly completed. This is a factor where a customer must use a large variety of colors or materials and does not desire to maintain large powder inventories.
- 7. Thinner continuous coatings can be applied. Coating thickness ranges for electrostatic spray are generally 3 to 30 mils.

### WHAT ARE PROCESS LIMITATIONS?

Several limitations common to both electrostatic spray and fluidized bed coating are as follows:

- 1. All parts require jigging or masking, since a holding point is needed. (Often the holding point is used simultaneously to mask the area not to be coated.)
- 2. Coating materials must be in powder form and carefully formulated to meet given application and service conditions.
- 3. The part substrate must withstand the necessary temperatures.
- 4. While these processes are theoretically intriguing and work well when done properly, a number of variables are involved. It is important to properly control these variables. Knowledge of these variables can enhance and greatly increase the versatility of the process. Conversely, overlooking one of these seemingly minor variables could cause serious production problems. It is quite important that a potential user work closely with someone knowledgeable in the entire process area to obtain a properly functioning production line.

### FLUIDIZED BED LIMITATIONS

- 1. Continuous coatings of less than 7 mils are difficult to apply.
- 2. The transfer time from the preheat oven to the dip tank must be kept to a minimum.
- 3. There are practical part size limitations to be considered, particularly where many different colors are involved.
- 4. Most successful users have found planning based upon a total system engineering concept produces best results. Designing a fluidized bed system is distinctly different from that required for other finishing methods.

### ELECTROSTATIC SPRAY LIMITATIONS

- 1. The disadvantages of the electrostatic spray process must be considered when contemplating use of this process. First, many of the coating powders on the market today are not suitable for coating cold parts or do not provide the desired film thickness on cold parts. Thus the advantages associated with a cold application of powder are lost to these materials.
- 2. Part configuration also limits use of the process. Parts with large differences in cross-sectional areas are difficult to spray cold and obtain a good coating. The coating on thin areas may degrade before the coating on heavy areas can fuse properly. Large parts that are to be preheated may lose too much heat during spraying. These parts are more easily coated via fluid bed where the part is completely immersed in powder within a few seconds after leaving the preheat oven.
- 3. Another difficulty arises when attempting to coat very sharp inside corners or inside holes or crevices (on hot and cold parts). Such areas are electrically shielded and the powder is not attracted into them. This usually results in incomplete coverage or pinholes.
- 4. Electrostatic spray produces more dust in the air than fluidized bed coating, so the potential hazard of dust inhalation by the operator is more severe. Proper dust masks and exhaust systems should be used. The spray guns are designed to provide a high voltage but low amperage charge to the powder which will prevent a dangerous spark from the gun. However, a spark still should be considered as a potential hazard. especially if the parts are not grounded properly.
- 5. Good coatings applied electrostatically may require a higher degree of operator skill especially if a hand gun is used. Uniformity of the coating, absence of pinholes in difficult areas, etc., are all largely dependent on operator skill.
- 6. A special problem unique to electrostatic spraying sometimes appears when spraying preheated parts. It is commonly known as "cobwebs". It shows up in the form of hairs or strings of plastics clinging from the coating.

Electrostatic powder spray technology is still in its infancy. New knowledge and techniques are rapidly being developed and the limitations mentioned here will undoubtedly soon be modified.

### WHAT PLASTICS POWDERS ARE COMMERCIALLY AVAILABLE?

The coating selector chart can be used to compare some of the important properties of the seven major available powders for fusion coating systems which include:

1. Vinyl for outdoor weathering, chemical and abrasion resistance and low cost. The durability and outstanding toughness of these vinyl finishes are of particular value in coating many consumer products. These coatings can also beused for general purpose electrical insulation having a dielectric strength of about 1,000 volts per mil.

After 10,000 hours' exposure in a Weatherometer, or in a salt spray cabinet, these melt mixed vinyl finishes are essentially in the original exposed condition except for slight loss of gloss.

The formulator has a wide latitutde in compounding these vinyl coating powders and in producing coatings with a wide range of properties. These powders are essentially composed of vinyl resins, plasticizers, stabilizers and pigments. There are, however, specific considerations, i.e., the formulator is not restricted to using resins which can be dissolved in solvents as in vinyl paints. All ingredients, must be carefully selected to withstand the heat of application.

In addition, there are two basically different methods of manufacture used in preparing these powders - "dry blending" and "melt mixing". The dry blended powders are available at lower cost than melt mixed formulations. This is because the ingredients are mixed dry directly with the powdered PVC resins. This is a short operation using simple and inexpensive plant equipment.

The melt mixed powders are produced by intensive milling of all ingredients while in a molten state. They are thoroughly mixed at elevated temperatures while molten under high pressure-shear conditions assuring complete dispersion of all components. This mixture is then reduced to powder by cryogenic grinding and selectively screened. Each particle of the melt mixed powders contains exactly the same amount of plasticizer, stabilizer, pigment and resin. These added plant operations which insure the superior quality of melt mixed powders also add cost. Further, a considerable plant investment in specialized machinery is required to produce these high quality melt mixed compounds. The improved properties they exhibit are primarily due to better dispersion and uniformity of all the ingredients, particularly the pigments. This is of importance in many applications such as outdoor weathering. Also, lot to lot variations are minimized which produces more uniform coatings as compared to dry blends.

There is a place for both dry blend and melt mixed coating powders. Each should be properly selected and used after a careful evaluation of the application requirements.

Dry blends are offered for lower performance requirements which do not involve long term outdoor weather exposure. They frequently are used to replace low cost plastisols.

Melt mixed vinyls are recommended for long term, high performance, end use requirements where characteristics such as outdoor weathering, salt spray or heat resistance, cathodic protection, detergent resistance, boiling water or hot oil immersion properties are important.

2. Cellulosic for outdoor weathering, abrasion resistance and decorative finish. These coatings are popular for decorative and protective applications. After 10,000 hours of exposure in a Weather-Ometer, cellulosic coated steel panels showed no loss of gloss or color and no evidence of edge corrosion.

- 3. Nylon for low frictional characteristics, wear and abrasion resistance. The combination of properties which has made the polyamide plastics unique for molded and extruded parts also recommend its use for coating applications. Nylon cannot be practically applied as a solution coating due to excellent solvent resistance.
- 4. Chlorinated polyether for high order chemical resistance. This relatively new plastics is receiving rapid recognition due to an excellent combination of mechanical, chemical, thermal and electrical properties. These coatings have excellent dimensional stability and are quite tough with good resistance to wear and abrasion.

Chlorinated polyether fusion bond finishes provide good electrical insulation even under high humidity and high temperature conditions and have very low moisture absorption. These coatings are adequate for continuous operation at 250°F and are in use to 300°F. This new plastics has excellent general chemical resistance and is widely used to coat equipment for the chemical industry. Coated cast iron parts have often satisfactorily replaced expensive high alloy steels.

- 5. Epoxies for electrical insulation. Epoxy fusion bond finishes demonstrate that both thermosetting and thermoplastic materials can be applied by this new coating system. Epoxy coatings have exceptionally good electrical insulation properties over a wide temperature range. They are also used in a wide variety of applications involving higher temperatures, appearance and toughness.
- 6. Polyesters are available in both thermosetting and thermoplastic formulations. The thermosetting polyesters are used primarily for electrical insulation and provide insulation at relatively high temperatures. The thermoplastic polyesters are used for outdoor weathering, abrasion resistance and electrical insulation. Though lacking in chemical resistance to mild alkaline solutions, this plastics resists mineral acids, salts and many solvents. The hard surface it provides resists dirt pick-up and scratching. Since primers are not used, proper cleaning and preparation of the surface to be coated is extremely important.
- 7. Polyethylene for low temperature chemical resistance and electrical insulation. Polyethylene coatings combine low water absorption and excellent chemical resistance with good electrical insulation. Polyethylene fusion bond finishes are used on food handling equipment, chemical processing equipment, battery holddowns and fans.

### WHAT KIND OF PARTS ARE BEING COATED?

Examples of the hundreds of different products now being coated in production included pipe, fittings, valves and other components which make up fluid conveyance systems for chemical processing plants; direct burial pipe; parts for dishwashers, refrigerators and washing machines; rotors, stators, bus bars, printed circuit boards and other electrical parts requiring insulation; distribution

transformer covers and tanks; outdoor fencing and selected components manufactured by or for the automotive, marine, farm equipment and construction industries.

In conclusion, several unique, versatile, powder coating processes are being widely used commercially to clad metals with plastics. Due to the comparatively heavy film which can be economically applied, it is possible to truly combine the superior characteristics of metal and of plastics into a single, functional part. For centuries, coatings have been considered only in terms of liquid materials. Now, both new and different plastics, as well as plastics in conventional use as coatings, can be applied to a wide variety of parts as a dry, solvent-free powder.

TABLE I

COATING SELECTOR CHART

	<u>Vinyl</u>	Cellu- losic	Ероху	Nylon	Poly- ethylene	Chlorinated Polyether	Thermo- plastic Polyester
CHEMICAL RESISTANCE							
Exterior durability Salt spray Water (salt, fresh) Solvents	E E E	E E VG	F VG G	F G F	F G VG	F E E	VG G G
Alcohols Gasoline Hydrocarbons Esters, ketones	E G P P	F G G P	E E F E	G E E VG E	E VG VG G F	E VG E VG F	E G P
Chlorinated Salts Ammonia Mineral acids	E E	VG P	E P	VG G	E E	E E	F G F
Diluteb Concentrated <sup>c</sup> Oxidizing acids Dilute <sup>b</sup>	E G E	G P P	E G G	F P P	E VG VG	E E	G P F
Concentrated <sup>c</sup> Organic acids Acetic, formic	G F	P P	P F	P P	P VG	G E	P G
Oleic, stearic Alkalies	E E	F F	E VG	VG G	VG VG	E E	G P
MECHANICAL AND PHYSICA					_		***
Abrasion resistance Flexibility Impact resistance Max. service temp., OF Dielectric strength	G E E 200+ VG	VG G E 180 VG	VG Fe G 350+ E	E G VG - 180 <sup>d</sup> G	F E F 160 E	VG F G 250 VG	VG F G 200 E
DECORATIVE PROPERTIES							
Color range Color retention Initial gloss Gloss retention	E VG VG G	E E E	P F G P	F VG G	G VG VG -	P G G	E G G F

Key: E = excellent; VG = very good; G = good; F = fair; P = poor

aThese data are intended only as a preliminary selection guide. Final selecttion should be made after consulting with coating formulator and after suitable testing. Data are based on CORVEL fusion bond coatings as supplied by The Polymer Corporation with the exception of the polyester.

bDilute = 10%

cconc. = over 30%

dup to 300°F in nonoxidizing environment

eRanges from good to poor, depending upon composition

### ROTATIONAL MOLDING OF HIGH DENSITY POLYETHYLENE POWDERS

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### INTRODUCTION

Of the materials currently available for rotational molding, high density polyethylene is receiving the most attention. High density polyethylene offers a broad range of desirable properties, including an excellent balance between stiff-ness and toughness, high resistance to heat and chemical attack, and greater resistance to creep or cold flow than is available with low density polyethylene. The design flexibility of rotational molding makes this material an ideal candidate for many consumer and industrial applications, which up to now have been non-plastics. The conversion to rotationally molded high density polyethylene is strikingly illustrated in three consumer products.

### TYPICAL APPLICATIONS

The first is a portable marine gasoline tank molded by Spartan Products, Inc., for Chrysler Outboard Corp. (Figure 1). High density polyethylene is replacing steel in this application. According to the manufacturer, a polyethylene tank is cheaper to produce and is more serviceable than steel, since it cannot rust.

Rotational molding was selected on the basis of styling. Blow molding was considered, but the design did not lend itself to this process. The tank features an integrally molded handle and two threaded openings for the filling cap and level indicator. In addition, four threaded inserts are molded into the tank to accept an injection molded escutheon plate.

The rear seat heater duct for the Ford Thunderbird is an excellent example of the intricate shapes with uniform wall thickness that are possible with rotational molding (Figure 2). This product is manufactured by The Wiremold Company, which found rotational molding to be the least expensive technique for this job.

The stiffness of high density polyethylene is utilized, along with its high heat resistance. The heater duct, subjected to an ambient temperature of 250°F for

four hours, showed no sign of distortion. A metal mounting clip is molded in the duct. The low stress level of the molding permits stapling or riveting without cracking. High density polyethylene heater and defroster ducts are replacing molded fiberboard and wire-supported fabric tubing; but more important is the fact that new engineering concepts can be put into practice with rotational molding.

A third item is a medium-priced phonograph case, molded by Moll Tool Plastics Corp. for Arvin Industries (Figure 3). Here again, rotational molding results in a lower cost than the original vinyl covered wooden cases. The assembly contains three rotational moldings - two speaker cabinets and a turntable base. The double wall configuration of the base cannot be duplicated by injection or blow molding. This unitized construction simplifies the assembly operation.

### MOLDING VARIABLES ARE OF PRIMARY IMPORTANCE

To utilize the unique design freedom that rotational molding offers and obtain the desirable properties from high density polyethylene, close attention must be paid to the molding variables.

In injection molding, blow molding, or thermoforming, the properties of the product depend primarily upon the characteristics of the resin. In rotational molding, however, this is not necessarily true. The ease with which the resin is molded, the tensile strength, and the stiffness will be determined by the resin type. But the toughness or durability of the product will depend upon how the resin is processed.

### MOLD HEATING TIMES, TEMPERATURES AND TECHNIQUES

The method of plasticizing is the basic difference between powder molding and the other processes for molding thermoplastics. Heat and pressure are used in other processes to melt and shape the resin. No pressure is used in rotational molding, only heat.

A variety of heating systems are being used in rotational molding. These include hot air convection, molten salt spray, circulating oil, infrared, and direct flame. Although they differ in efficiency, complexity, and flexibility, the principle of plasticizing is the same, i.e., the powder is melted through contact with a heated mold surface.

In molding low density polyethylene, the heating is normally stopped at the point where all of the included air pockets have been removed from the molten part. If carried further, embrittlement would result from thermal degradation. With high density polyethylene, which has greater heat stability, the heating period can be extended. This extended heating removes all traces of particle boundaries, giving a higher degree of fusion.

The effect of heating time on impact strength is illustrated in Figure 4. The resin is a 0.95 g/cc density, 5.0 g/l0 min. melt index polyethylene powder. A series of 1/8" thick parts was molded at the indicated heating period using molten salt spray equipment at 550°F. Flat panels were cut from each molding and tested with a constant height, falling weight impact tester at -20°F. The program was repeated using parts molded at 500°F to show the influence of molding temperature.

You will notice that the impact strength can vary by a factor of 5 with parts

molded from the same resin. The tensile strength and flexural stiffness are essentially the same for all parts in this series.

These curves show that for each molding temperature, a point exists where the impact strength is maximum. To the left of this point, incomplete fusion is found; to the right, polymer degradation begins to have an effect. The two minute molding looked quite satisfactory from the outside, but the inner surface was rough with small air inclusions. The maximum toughness for this resin and wall thickness occurred at a heating time of 8 minutes with a heating temperature of 550°F. At a lower temperature, the curve is shifted to the right, but the magnitude is not affected. The amplitude of the curve will depend upon the resin type. The location of the curve on the time axis will vary with each rotomolding machine and mold construction.

Although this curve represents a particular hot salt machine, similar curves have been obtained from hot air equipment. With one commercial hot air machine, an oven temperature of 850°F was found to be equivalent to this 550°F salt temperature curve. It appears that hot air convection transfers the heat at a lower rate, but brings the mold up to a higher temperature, which results in the resin absorbing the same quantity of heat in an 8 minute period.

For any given resin, mold and oven, the heating period will depend upon the desired wall thickness of the part being molded. The size of the part is considered irrelevant to this study. Figure 5 compares low density with high density polyethylene and shows how the required heating time is affected by changes in wall thickness. Once a semi-molten layer of polymer is obtained inside the mold, the heat transfer rate is dependent upon the polymer's thermal conductivity. As might be expected, the curves are essentially straight lines. In general, if the wall thickness is doubled, the heating period should be doubled.

The point at which the curves cross the time axis is considered to be the mold heat up, or induction period. Heavier or thinner molds will shift the curve, but should not change its shape.

Although the thermal conductivity of high density polyethylene is greater than low density, it requires about 40% longer heating time to mold. This is due to the higher softening point and the fact that extended curing was used with high density polyethylene.

In going through the rotational molding process, the molecular weight, or melt index of the polymer is altered as illustrated in Figure 6. Each point on the graph represents a separate molding at a wall thickness of 1/8". The resin is a nominal 5.0 g/10 min. melt index, 0.95 g/cc density, non-stabilized polyethylene. As the heating time is increased, the melt index drops to a minimum, or the molecular weight reaches a maximum. If heated long enough, the polymer begins to "crack" and the melt index increases. This change in melt index coincides with the change in impact strength in Figure 4. The point where the melt index reaches a minimum corresponds to the maximum toughness in the molded part. It is evident, therefore, that melt index can be a useful laboratory tool in determining the optimum molding conditions for any application.

It is believed that the decrease in melt index is caused by a combination of three factors. High density polyethylene consists of carbon chains of various lengths. The shorter molecules are needed for easy processing. The long chains give the polymer toughness. During the heating process, some of the short chains are "boiled" out of the resin, leaving behind the heavier molecular weight fraction.

When the part is cooled, this gas, which has combined with oxygen in the mold, condenses on the inside surface of the part to form an amber colored film.

To prove this theory, low molecular weight polyethylene was purposely added to a standard resin. The oxidized film found on the inside of a part molded from this blend was noticeably darker than the sample molded from the standard resin.

In addition, it is thought that some thermal crosslinking occurs during the extended heating period. That is, recombination or branching of thermally cracked chains into heavier molecules takes place.

Another factor, contributing to a lower melt index, is oxygen pickup in the form of carbonyl groups.

Regardless of the cause of this change, a low melt index signifies a well fused part with the maximum impact strength obtainable from that particular high density powder.

The brownish cast, which is found in well cured parts, has been objectionable for many non-colored decorative items, such as mannequins, light globes, beverage containers and appliance components. To meet appearance requirements, custom molders have been forced to cut back on the heating time, thus sacrificing physical properties.

### PURGING THE INTERIOR OF ROTATIONALLY MOLDED PARTS

The most obvious way to eliminate discoloration and odor is to remove the oxygen from the mold. This can be accomplished by flushing the air out of the mold with an inert gas, such as nitrogen or carbon dioxide. The piping arrangement for inert gas purging is schematically represented in Figure 7.

The mold is purged before the heating process is started. The gas flow rate will depend upon the size of the mold. A flow rate of 10 to 15 cfm for 30 seconds is sufficient for a mold volume of 1 cu. ft. If the gas flow is continued during heating it tends to have a cooling effect on the resin, and upsets the previously established heating time. As the mold heats up, a pressure is created inside the mold, which keeps air from returning. The inert gas is turned on again during cooling. A low molecular weight film will still be present on the inner surface of the molding, but will add no color or odor to the part.

Impact strength and environmental stress crack resistance are slightly improved by the use of inert gas. Table I shows the results of tests performed on samples molded at identical conditions except that a nitrogen purge was used on one set of samples. The impact strength was measured with a constant height, falling weight tester at -20°F. Test method ASTM D 1693-60T was used to measure stress crack resistance. The controlled imperfection was omitted to compensate for the high stiffness of the 0.95 g/cc density polyethylene.

Although these tests do not show a significant difference that would justify the use of inert gas on the basis of physical properties, they do indicate that no adverse effect results from its use.

The problem of odor and discoloration is also being approached from a resin standpoint. Development studies are currently under way to reduce the polyethylene vapors inside the mold by using stabilizers and by changing the molecular weight distribution.

### EFFECTS OF COOLING RATES AND TECHNIQUES

In the investigation of heating, a single rate of cooling was used. If the heating rate is held constant and the cooling varied, the appearance of the molding will be affected, as well as its physical properties.

Crystallinity in high density polyethylene can be as high as 95%. The cooling rate affects the amount of crystallinity and the shape of the crystallites as the polymer solidifies. Slow cooling produces a highly crystalline product, high in density and stiffness, but more brittle. Rapid cooling tends to reduce the size of the crystals, giving a tough, more flexible part, with greater resistance to stress cracking.

Table II shows the results of tests performed on a single part, which was cooled at two different rates. A rotationally molded sample was removed from the oven and cooled to a temperature of about 225°F. The mold was then opened and the sample cut in half. One half was quenched in a water tank; the other half was left in the air to cool.

The quenched section had considerably lower density, or crystallinity, than the air cooled section. The impact strength was only slightly higher, but a significant difference was found in the stress crack resistance. In measuring stress crack resistance, the boiling water conditioning of ASTM D1693 was omitted, in addition to the controlled imperfection, so that the effect of cooling stresses could be observed.

High density polyethylene parts are generally cooled with a highly atomized water spray on the rotating mold. To obtain the best physical properties, rapid cooling is desirable. However, if the water is applied too vigorously, the part may shrink away from the mold, thus substantially reducing the heat transfer in certain areas. Warpage or localized brittleness may be the result. In practice, control of warpage dictates how fast a part can be cooled.

One way to obtain rapid and uniform cooling is to use a combination of external and internal cooling. Cooling the polymer from both sides not only increases the rate, thus improving the physical properties, but gives better dimensional control by promoting uniform shrinkage. The same piping arrangement shown in Figure 7 for inert gas may be used for internal cooling. Cooling time can be cut in half by passing chilled air or water vapor through the mold.

Liquid carbon dioxide is another possibility for internal cooling. This process has been successfully used in blow molding. As the liquid passes through an orifice in the mold, it changes phase and absorbs heat from the hot polymer. A thin wall part can be cooled in a matter of seconds by this method.

Precision moldings can be obtained by using air pressure to hold the polymer against the mold while it is cocled. Although rapid cooling is possible with the "roto-blow" technique, it has the disadvantage of freezing shrinkage stresses into the part. High quality mold parting lines are required to prevent flashing. A pressure of 1 to 15 psi is normally used.

Shrinkage is unusually hard to predict in rotational molding. High density polyethylene will shrink from 1-1/2 to 3-1/2%, depending upon the density and melt index of the resin, the shape of the part, and the release characteristics of the mold. The most significant factor, however, is cooling. As Table II shows, the highest shrinkage is found in slowly cooled parts. This is to be expected, since

the molded density is higher. Parts with an interrupted surface tends to shrink less than smooth flat parts. The shrinkage on one long slender tank with transverse corrugations was 1.85% over the long dimension. At right angles, or parallel to the corrugations, the shrinkage was 2.4%. For mold construction purposes, a shrinkage factor of 2-1/2% is normally used for high density polyethylene.

### REFERENCES

1. Duncan, R.E., et al, "How to Buy Rotational Molding Machinery", Plastics Technology, June, 1965, p. 95.

TABLE I

EFFECT OF INERT ATMOSPHERE

### 0.950 Density, 5.0 Melt Index PE

	Nitrogen	Air
Impact strength (Gardner), ft_lbs/0.125 in. at _20°F	40	37
Stress crack resistance, hrs/F50	2.9	1.9

### TABLE II

### EFFECT OF COOLING

### 0.950 Density Polyethylene

	Fast	Slow
Density, grams/cc	0.949	0.955
<pre>Impact strength (Gardner),   ft_lbs/0.125 in. at -20°F</pre>	35	33
Stress crack resistance, hrs/F50	> 36	$14\frac{1}{2}$
Shrinkage, %	2.4	3.2



FIGURE 1: Portable marine gasoline tank, molded by Spartan Products, Inc. for Chrysler Outboard Corp.

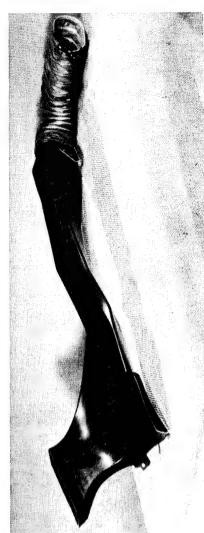


FIGURE 2: Heater duct for Ford Thunderbird, molded by Wiremold Company

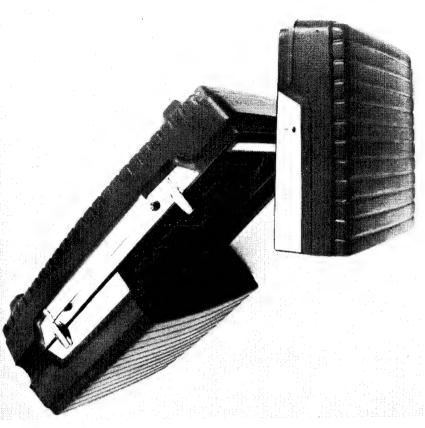
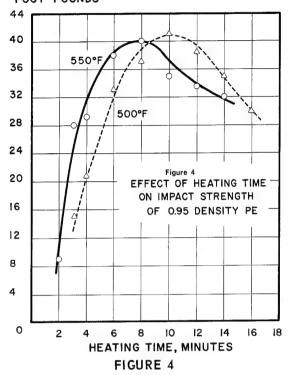
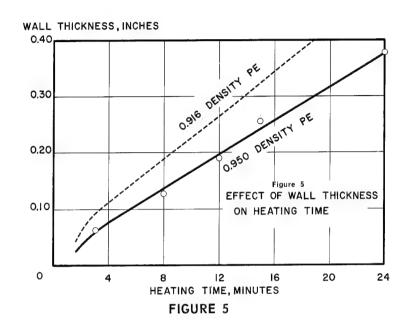


FIGURE 3: Three piece stereo cabinet molded by Moll Tool and Plastics Corp. for Arvin Industries

### GARDNER IMPACT FOOT-POUNDS





### MELT INDEX GRAMS/IO MIN. 6.0 5.0 Figure 6 EFFECT OF HEATING ON MELT INDEX OF 0.95 DENSITY PE 4.0 3.0 500°F 2.0 1.0 550°F 0 24 12 16 20 HEATING TIME, MINUTES

FIGURE 6

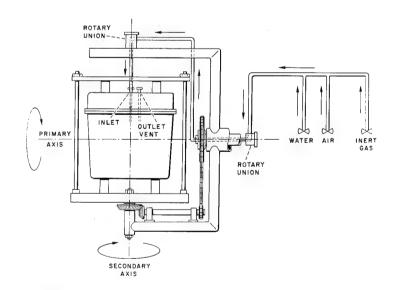


FIGURE 7: Schematic of rotational molding mechanism for inert gas purging and internal cooling

9622-8

### ROTATIONAL CASTING OF POWDERS

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Industry experts predict that by 1970 the volume usage of rotational casting powders will be twenty times the amount used in 1965. Last year alone, new equipment sales increased 300%. In the last five years advancements in materials, technology and equipment have surpassed those of the previous twenty years. Impact resistance, weatherability and cycle times have all shown a tremendous improvement. Product emphasis has switched from toys to containers and industrial and automotive applications. These advances are paralleled by corresponding improvements in equipment capability which permit larger products and faster cycles. Machines now will cast parts up to seven feet in diameter and eight feet in length.

The basic principles of partially filling a very economical, hollow mold with liquid or powdered material, heating this material while rotating the molds in two perpendicular planes and then cooling the material to solidify the cast part, remain the same as they have for the last twenty-five years. This sounds very simple and basically it is; however, both technique and equipment have become much more sophisticated than was envisioned even two years ago.

Equipment is available today which is almost completely automatic. Once the controls are programmed, the operator need only touch three loading station positioning switches. Physical labor has been reduced to undoing the mold clamping arrangement, removing parts and reloading and clamping the molds. The molds are automatically transferred to the oven and cooling chamber (Figure 1). To date, emphasis has been placed on equipment of maximum versatility. Relatively little time has been devoted to methods of clamping and automatic mold handling. There is considerable room for improvement in this area; however, equipment is now in the planning stage which may permit automatic mold handling, clamping and part handling while, at the same time, further shortening cycles.

Early in our experience it was determined that it was not only necessary to be able to adjust the speed of rotation to match the different viscosity materials but, in addition, the old standard 4 to 1 ratio was no longer accurate enough for today's sophisticated applications. It was found that each basic shape had a ratio of number of times the mold turned over to the number of times if turned around that would fill the mold the most uniformly. With proper speed and ratio of rotation, part wall tolerances of  $\pm$  .005" are easily maintained regardless of the part shape.

As an example, one gasoline tank, which in side view looked roughly like a trapezoid and was approximately four feet long, was first tried on a 4 to 1 ratio.

The ends of this part were approximately .045 thinner than the rest of the tank. An electrically controlled change to a 2 to 1 ratio immediately resulted in the wall thickness of the ends becoming the same as that of the rest of the part. However, a distribution problem developed in a different area. Because of this problem, the ratio was changed to 3 times over to 1.2 times around and an overall wall tolerance of  $\pm$  .006" was maintained on all future parts. Other radical changes from a standard 4 to 1 ratio have resulted in equally even walled products.

The number of molds on each side of each standard arm may vary from one to fifty cavities. Where operations utilizing molds of radically different shapes or parts made of materials requiring radically different heating cycles are encountered, each separate arm or spindle of molds may be individually adjusted for rotating speed, ratio, oven residence and cooling cycle. This permits different shapes and materials to be run on each individual arm. Oven residence times control the total cycle and, for forced air machines, may range from two to twenty minutes depending upon the material and thickness of part wall. The average production cycle will be approximately seven minutes. The total of the oven residence times gives the total number of complete 360° indexes per hour. Therefore, approximately every seven minutes you might expect to remove from 2 to 100 parts. Various combinations of water and fan cooling may be programmed for each arm to insure that each different material will attain maximum physical properties.

Although polyethylene and polyvinyl chloride remain the major usage materials, improvements in these materials and the addition of new materials are continually opening new markets. Examples include butyrate and proprionate for lighting, furniture and store window mannequins; polyethylene for industrial battery jars, containers and gasoline tanks. A few short years ago, only low or medium density polyethylenes were available; now polyethylenes which are as low as 1 melt index and up to 0.962 density, are run in production. Thermally crosslinkable polyethylene is also available. Ethylene-vinyl-acetate is replacing some other materials in toys, traffic cones and other applications where flexibility at extremely low temperature is desired. The rapid development of powdered vinyl is finding increased usage in automotive and other commercial foamed skins. Other materials such as rigid powdered vinyl, polycarbonate, ABS, styrene, polystyrene, polypropylene and combinations such as result in polyethylene skins with expandable polystyrene cores have been cast experimentally, but they require further development before becoming available for production usage. Parts have also been experimentally cast from fluoroplastics, nylon, polyphenyleneoxide, and acetal resins.

In other systems of forming parts, the material is plasticized in an extruder at some point before it is formed into its final shape. After molding, it is restrained in this shape until partially cooled. Consequently, changes in melt index, density and particle size are somewhat nullified by the pressure of the extruder screw. The utilization of pressure to force the polymers into shape has an undesirable side effect in that it tends to build stresses into the parts.

Rotational casting utilizes no pressures to plasticize material or to form a part. The flow of material, inside the mold, is controlled entirely by gravity. Centrifugal force is carefully avoided in normal rotational casting techniques in order that pressure does not modify the material distribution. The plasticization of the polymer is accomplished solely by heat; hence, changes in melt index, density and grind will greatly change the flow of the material and will also affect the molding cycle.

Normally no pressure is used to hold the part against the mold during cooling; therefore, the rate of cooling and the mold release used may alter the size of the finished part. This method of casting and cooling results in stress\_free

parts which perfectly reproduce the mold surface detail. Thus, wood grain, leather grain, or mirror finish parts may easily be obtained. Rotationally cast articles will not try to change their shape back into a sheet, parison or mass when exposed to heat. They will try to retain their cast shape until subjected to temperatures sufficient to start to melt the polymer.

Let me illustrate a few actual problems encountered in rotational casting today. A company had been manufacturing decorative indoor light globes by blow molding the globe, spraying this globe with glue and rolling it in transparent or
opaque colored pellets. I am sure all of you have seen similar globes in stores.

Problem #1 was how to permit practical runs. Problem #2 was how to eliminate several expensive hand operations. Problem #3 was how to attain uniformity in the basic
globe. Problem #4 was reduction of scrap.

It was believed that these problems should easily be handled by rotational molding. A mold of a finished pebbled surface globe was obtained at a cost of considerably less than \$500, including patterns. Parts of even wall thickness were run with no scrap. However, one small problem developed! Because of the hundreds of undercuts caused by the simulated pellet covered surface, we could not open the mold to remove the part! Needless to say, this was considered a rather time-consuming and expensive hand operation. We then decided to concentrate on problems #3 and #4. Using a plain globe mold, parts of  $\pm$  .002" were run with no scrap or trim. 100% of the raw powdered material was returned in the finished part.

This was fine as far as it went, but, admittedly, was not particularly earth—shattering in result. However, other work we were performing at this time was to later solve these remaining problems. Another company was struggling with wall thickness uniformity involving butyrate store window mannequins. The rather crude, experimental grind, butyrate particles had tails on them which hooked together during the casting. This resulted in parts with considerable bridging and large agglomerates of material on the inside part wall. Because the finished part had to show a uniform texture, this effect was completely undesirable. After considerable experimentation, butyrate pellets were added to the powdered material. Theoretically, the pellets, being much larger in size than the grind, would take longer to attach to the part wall and, therefore, would remain free to rattle around the inside of the piece and break up these agglomerates. It was determined that adding from 25% to 50% pellets to the grind did, indeed, prevent the formation of the rough masses. The part shown in Figure 2 is the first one successfully rotationally cast without agglomerates. Notice the relative evenness of the part walls.

An unexpected bonus was obtained as sufficient heat was applied to the mold to fuse the pellets into the part wall thus adding to its strength. This new technique solved the problem of agglomeration without increasing the total amount of material used. Improvement in materials and grinding technique has since greatly reduced the tendency for ground butyrate to form into masses; however, in this particular mannequin application, pellets are still being added to the powder.

In order to observe the effect of the pellets on the finished part, the same technique was tried on other types of materials and products, including the light globe. Butyrate is a desirable light globe material due to its temperature resistance and weatherability. The first trials utilized a clear butyrate powder with transparent colored styrene pellets. Because the styrene did not mix with, but only bonded to, the butyrate wall, the pellets gave the same effect as the original rolled pellet globe, but with the added benefit of eliminating all of the hand operations. Moreover, the consumer liked the fact that the pellets were on the inside of the part thereby making the globe considerably easier to clean.

Following experimentation with various colored styrenes, transparent colored butyrate pellets were obtained. Because this material became a homogeneous part of the wall, these globes had a uniquely different appearance. Thus, the remaining light globe problems were resolved through development of both new materials and process techniques. Utilizing basically this same system, another molder has concentrated on industrial and street lighting with excellent results.

A far different problem was incurred by a company trying to mold a large metal support plate into a liquid PVC seat skin. Speeds, ratios of rotation, cycles and practical production methods were soon determined; however, the customer was still not satisfied because approximately 1/2 pound of material per part was wasted when it jelled on the metal support plate molded into each part. This material also interferred with the bond between the support plate and the urethane foam which was, subsequently, inserted into the skin. By changing from liquid to powdered vinyl, parts of a uniform wall and an excellent mold definition were obtained. The skins had a consistently smooth inside surface with no runs or "button hooks". An added benefit was the attractive advantage in basic material cost and the reduction of cycle time by two minutes. Due to the fast cycle, the insert did not become hot enough to cause any of the powder to adhere. Consequently, a further cost saving as well as a strong foam bond was obtained. In some automotive applications, production cycle times of as little as two minutes are achieved.

Molders and material suppliers continually use our laboratory facilities for development of new products and materials. As a result, we have participated from the very first in development of such items as automotive and marine gasoline tanks. In these instances, it was first necessary to work with the material suppliers to develop a completely new technology and type of material which would "thermally crosslink" before we could manufacture tanks which would even begin to meet automotive specifications.

Problems were encountered in impact resistance, a mount of additives, cycle times and cooling. Atypical patterns of material distribution resulted from the prolonged exposure of polyethylene to high temperatures and necessitated mold design modifications. The automotive industry had to be educated in safety features, design freedom and favorable economics. Much was learned regarding the effect that rate of cooling had on the density and impact resistance of the finished part. The best mold temperatures and oven heat input rates had to be determined. One of the most annoying problems was the relatively great shrinkage of high density polyethylene. If this shrinkage occurred unevenly, a warped part would result. If the part was allowed to shrink too fast, it separated from the mold too soon, thereby prematurely ending the transfer of heat out through the mold wall. The part would "oil can" and cool too slowly for optimum physical properties. If it did not shrink rapidly enough, stresses set up in what should be a stress-free part.

This work gave new insights into types and application of suitable mold release agents which would permit consistently warp-free, high density parts. It was determined that dry-blending approximately 1/20% of calcium or zinc stearate powder into the material was sufficient to produce good parts. Experimentation is continuing along these lines but, to my knowledge, no other release agents have yet been discovered which will give consistently better results in production. In sufficient quantities, stearates may become an embrittlement agent, but in the concentrations currently being used, no significant embrittlement has been found.

Oven residence times required to "thermally crosslink" certain high density polyethylenes are approximately twice those required for normal fusion. These times may vary from nine to thirteen minutes depending on the mold, part wall thickness and oven input temperature. Rapid cooling is important to bring the

temperature down from approximately 475°F to less than 100°F in order to obtain the smallest possible crystal growth. Proper application of mold release will cause the part to stick to the mold wall long enough to accomplish the desired amount of cooling and still permit easy removal of a distortion and stress\_free part.

Tanks have been roto-cast which were tested on a 5/8" nose Gardener Falling Weight impact testing. Impact resistance of up to 400 in.-lbs. at -20°F were reported from 1/8" thick samples.

In our laboratory, tests were conducted with a free-falling weight having a l" nose. This weight was dropped from a constant four foot height at 70°F. Results of up to 600 in.—lbs. were obtained from 1/8" thick samples. It was interesting to note that much higher impact resistance resulted from the weight striking the normal outside of the part than could be obtained when the section was turned over and struck on the inside wall. This indicates that the material next to the mold may have started to degrade. Care should be taken to utilize molds of sufficiently good heat transfer to be compatible with oven cycles which will minimize degradation.

Considerable excitement has been generated recently over the merits and demerits of inert gas injection prior to the heating cycle. Time does not permit a full discussion of this process, but I would like to state that in certain instances this looks like a very practical way to reduce the oxidation odor inherent with overcured polyethylene. Equipment to inject inert gas or internal cooling gas into the molds may usually be incorporated with minor modification of present machines. For parts with walls of greater than .250 thickness, it is desirable to add internal cooling to the normal external cooling in order to minimize resin crystallinity and part warpage.

I have mentioned only a few of the problems encountered in "thermal cross-linking" materials for applications such as gasoline tanks. Proper programming of equipment to meet these and other problems is important in order to achieve and maintain optimum physical properties. Only a slight variation in heating or cooling may have a pronounced effect on the final part. During the prototyping of one gasoline tank, tests were run in which only the oven residence time was changed; all other factors remained the same. The following impact test results were obtained from a 5/8" nose Gardener Impact tester. 1/8" thick samples were tested after prolonged exposure to -20°F temperatures.

It can be seen in Figure 3 that "thermal crosslinking" technology can be somewhat touchy. For this reason, it is suggested that competent assistance be sought by anyone contemplating the manufacture of "thermally crosslinked" products.

Care should be exercised to select materials having properties best suited to the needs of the product and not vice versa. Apart from the more obvious choice of the general material type, changes in such things as additives and grind may affect the product and cycle. Probably the most popular particle size is a thirty-five mesh grind. This is a good general purpose size; however, on items such as containers with no surface detail or relatively simple toys where price is more important than surface finish, approximately 3¢ per pound may be saved by using a sixteen or twenty mesh powder. On the other hand, parts which are highly detailed or have a wood grain or mirror finish may require a fifty mesh material to achieve the desired quality.

Consideration should also be given to proper choice of molds. The type of mold should be determined by the material and end product. For instance, the plasticizer contained in PVC serves as an excellent mold release. Since vinyls are

usually flexible, especially when warm, intricate parts may be removed through relatively small openings. Electroformed molds lend themselves ideally to this type of casting and are utilized for such items as toys, auto sunvisors, seats and armrests. The hard nickel inner mold surface will give almost any desired surface finish and will last a long time with relatively little attention.

Aluminum or steel molds may be used for parts cast from materials such as polyethylene, which requires mold release and may be expected to give the molds harder wear. Intricately shaped or detailed parts are usually formed in 1/4" wall cast aluminum molds.

Molds which have been cast in plaster usually have a smoother, less porous surface than those cast in sand. These cast molds give excellent detail reproduction and have good uniform heat transfer.

Molds for containers having no surface detail may be economically obtained from spinnings and fabrications. These are by far the most economical type of molds and are in general use in the container industry. Other types of products made from fabricated molds are traffic cones, photographic developing tanks, and water softener components.

We have endeavored to present the current state of the art of rotational casting along with some typical development problems and their solutions. We sincerely believe that this "art" will continue its rapid growth until it becomes a science.

### ACKNOLWEDGMENTS

The author wishes to thank the following companies for their assistance:

Astron Corporation, L. A. Darling Company, Formed Plastics, Inc., Eastman Chemical Company, Fulton Manufacturing Co., Inc., Hamilton Cosco, Inc., Phillips Chemical Company, Sun Corporation and U. S. Industrial Chemicals Company.

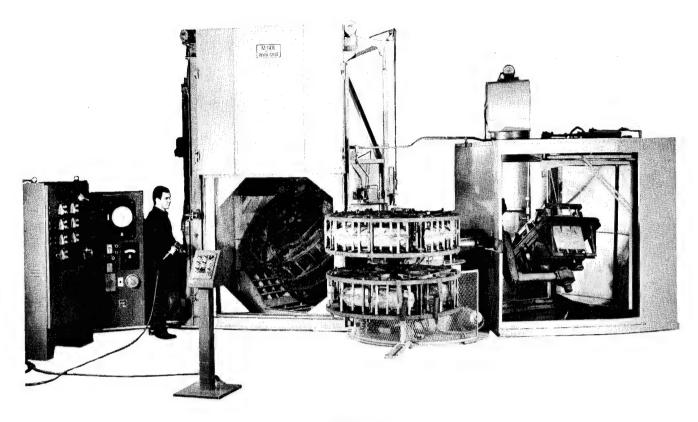


FIGURE 1

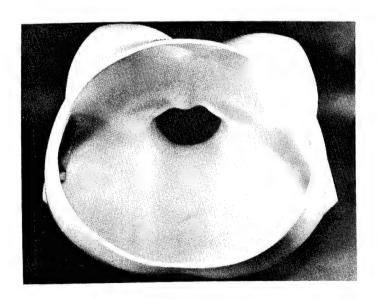


FIGURE 2

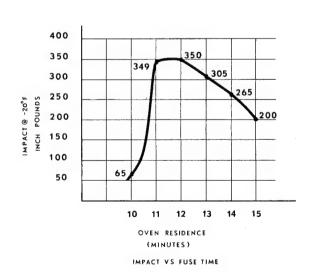


FIGURE 3

quadra 1724

### POWDERS FOR ROTATIONAL MOLDING PROCESSES

Robert Chandgie

Fusion Rubbermaid Corp.

Statesville, N. C.

The material requirements of rotational molding probably demand more attention than in any other form of plastics process. Basically, the long cycle times, high temperatures, lack of pressure and the presence of oxygen during molding, tend to tremendously reduce the effectiveness of the standard available materials.

The time-temperature cycle in rotational molding, being of long duration with high temperature readings, reduces the strength of the standard polyolefin materials by crystallization and, thereby, render the finished product weak and of poor quality. We feel the powdered material should be capable of withstanding this high heat, long cycle time duration without undue harm, and offer products with good impact and tear strength qualities.

Since there is no pressure exerted in rotational molding, the powdered material must flow into all areas of the mold cavity and thus adds to prolonging the cycle time. The nature of this process requires fusing of powdered particle to particle, without pressure; it is a matter of time related to temperature as shown in Figure 1. This figures shows the wall gauge of a typical part as a function of length of time mold is rotated in a  $650^{\circ}$ F oven.

Oxidation is another factor which is much more prevalent in rotational casting than in any other form of molding and plays a tremendous role in affecting the qualities of the finished product. We feel the material formulation should not be adversely affected by oxidation in order to offer superior product quality.

Now that the desirable features of rotational molding powders have been outlined, we must determine how to prepare these materials. Specifications must be written to prepare powdered formulations which will allow the finished product to perform as was intended. The correct additives must be used which are compatible with the base resin. These additives, such as, impact modifiers, anti-oxidants, and heat stabilizers, should be added to prevent material failure while molding and to assure long service life of the finished produce. Figure 2 illustrates the effects of additives on stress cracking.

If the customer's product requires color, the correct pigment selection is essential to prevent fading as this is also an important quality element. Some available plastics color pigments or concentrates do not lend themselves to the rotational molding process and, therefore, should not be used.

The base resin selection is most important as it is the foundation and must be selected to fit the product performance. For rotational casting we feel the base resins must be specially tailored in the polymerization process and not blends or

standard resins intended for other molding applications. These special base materials should be prepared to offer good melt flowability, which is required in rotational molding, should resist the tendency to sag or run as may be experienced in thick wall sections, and should be of the correct melt index and density to fit the specifications. In other words, the base resin must be a narrow specification, first line material and, in all cases, should be specially prepared for powder processing. This is very critical, and if not achieved the final powder will not be successful.

After the correct base resin has been secured, the additives tested, and the proper color pigment selected (if required), the material is ready for compounding. This process step is equally important because, if performed incorrectly, the material will not pass the specifications. We have always felt that the superior performance in colored materials for rotational molding could only be obtained by compounding the color into the mixture. Even small amounts of pigment to obtain the slightest tints can adversely affect the performance of the final product. See Figure 3 for the effect of this on impact. Color streaking, pigment deposits and the inability to attain color uniformity are quite common with dry-blending of materials. It has also been found that plastics to pigment fusing of particles rather than plastics to plastics particle fusing occurs in dry-blended materials and greatly reduces the strength of the final product. The slightly higher cost of compounded materials related to dry-blended is normally offset by the ability to attain color uniformity, lower scrap rate and less product failure. If all the intangible factors can be added, the compounded material is always the better buy.

The mesh size and uniformity of the powdered material is also very important as this has been found to offer better surface quality, aid in molding, help the strength characteristics, and reduce the scrap rate. We feel the ideal powder profile is to grind all the material through a 30 to 35 mesh screen with as small a percentage possible finer than 80 mesh. This profile was selected through many trials of mesh size performances. There are certain products which may require different profiles due to their particular design irregularities.

Most of our efforts to date have been with low and high density polyethylenes and the polyethylene copolymers. Our laboratory has evaluated other materials for rotational molding and feel, with time and technical advances in both materials and equipment, a number of other powders will lend themselves to this process. Liquid vinyls (plastisols) have been rotationally molded for a number of years with great success, but due to demand for products which will stand higher temperatures, possess greater strength, and have less warpage and shrinkage, new materials will enter. Some of the materials considered are difficult to process and grind, and new techniques will have to be developed to correctly handle them. Some exciting new advances have been made recently and materials never before considered applicable to rotational molding may now be used.

In Figure 4 the influence of molecular weight distribution and compound modification on impact strength is shown and points to possible improvements in future compounds.

The number of rotational molders has grown quite fast in a few short years and powdered material requirements have increased. With this growth, however, has also come a demand for improved materials to fit the product needs. With advances in equipment, powders will have to keep pace to insure faster cycles with higher quality and performance. There is absolutely no doubt that there is a real need for rotational molding; and it has been found that in the production of many items this process is quite competitive with blow molding, injection molding and thermoforming. Powder molding offers the production of some items that were impossible to mold by

any other technique.

There are at present powdered materials to meet the requirements and new powders will be developed to keep pace and offer the right materials to meet new product applications. Considerable interest in the powder molding process has and is being shown by major companies in varied industries ranging from automotive to toys. With additional refinement in equipment, techniques, and powders, there is almost no limit on future product applications for rotational molding.

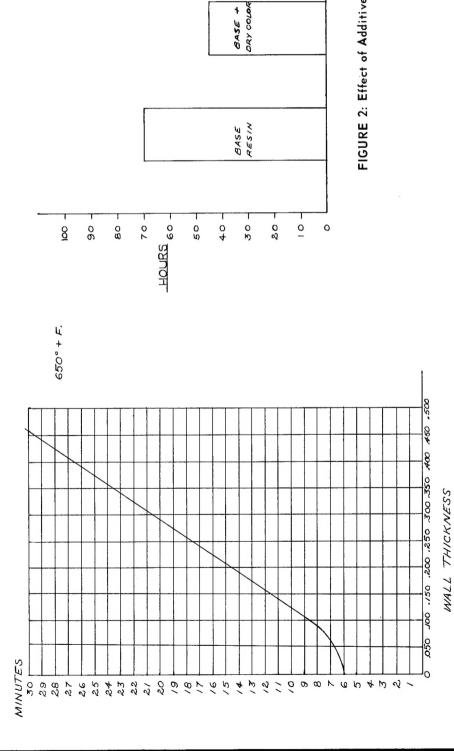
ROTATIONAL MOLDING TIME—TEMPERATURE CURVE

POLYETHYLENE MATERIALS

1000 HOURS -NO FAILURE

50°C IGFPAL 630

STRESS CRACK



SPECIAL
COMPOUND
COLOR +
MODIFIERS

FIGURE 2: Effect of Additives on Stress Cracking

FIGURE 1

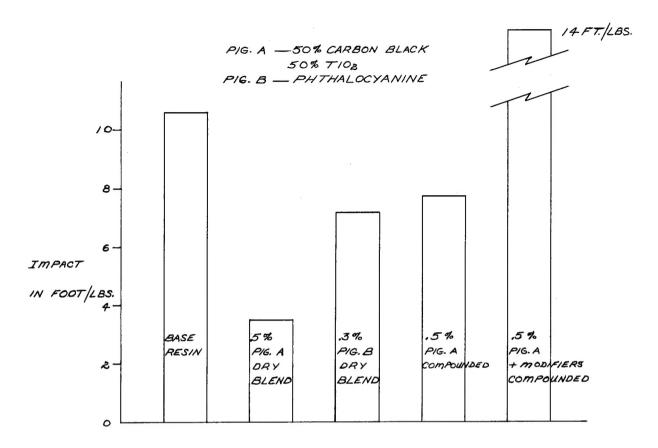


FIGURE 3: Effect of Pigments on Impact

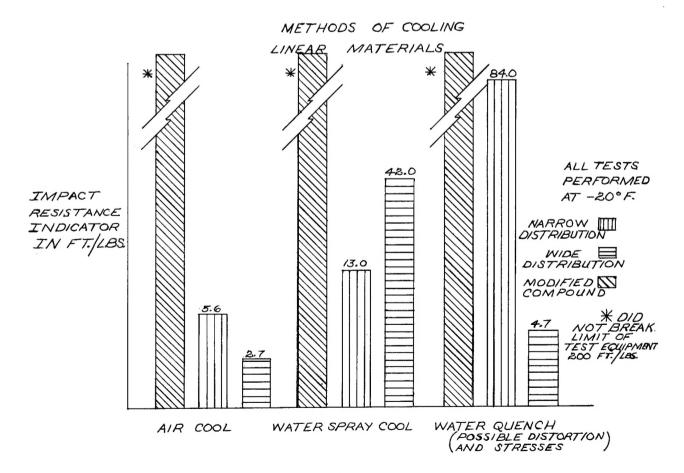


FIGURE 4

### INITIATION FEE MUST BE ATTACHED FOR PROCESSING.



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### MEMBERSHIP APPLICATION

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I certify that the statements	made in this application are correct.	I agree, if elected,	at qualifying years of exp	erience.	
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## COMPLETING THE APPLICATION

## Grade of Membership.

Membership grades are based on experience credits which are earned as follows:

## 1. Experience credits earned for education.

5 credits Doctorate in science or engineering subject: Bachelors in science or engineering subject: Masters in science or engineering subject:

Other degree in non-science or non-engineering subject:

When filling in the "Statement of College Work" on the reverse side of this application, please place the corresponding number of credits earned Maximum credits allowable for education shall be six (6). in the right-hand column.

the engineering skill required for each position to 2. Experience credits for qualifying experience in plastics or plastics engineering are earned at the rate of one (1) per year, e.g. 51/2 years of qualifying experience = 5½ credits. Please detail carefully help the Credentials Committee judge experience as "qualifying." When filling in the "Record of Qualifying Experience in Plastics" on the reverse side, please place the amount of time spent in each position (in years and months) in the right-hand column. When you have determined the number of credits which you believe you have earned consult the following membership grade requirements. Indicate on the reverse side the grade of membership for which you believe you are qualified.

GRADE	REQUIREMENTS
Senior Member	Minimum of twelve (12) experience credits and maintained continuous membership in the Society for a minimum of two (2) years.
Member	Minimum of six (6) experience credits
Affiliate Member	Less than six (6) experience credits
Student Member	Regularly enrolled student (full- or part-time) in a course of study in plastics and between the ages of 16 years and 26 years, inclusive.

## THIS PORTION MUST BE COMPLETED FOR PROCESSING OF YOUR APPLICATION.

Please check off the principal activity of your company under either Manufacturing or Non-Manufacturing.

### MANUFACTURING

- 1. 🗌 Electrical & Electronic Machinery, Equipment & Ap-
- 2. 

  Motor Vehicles and Equipment
- 3. 

  Transportation Equipment (except Motor Vehicles)
- 4. ☐ Professional, Scientific and Controlling Instruments, Photographic & Optical Goods, Clocks
- Iron, Steel & Nonferrous Metals & Machinery (except Plastic & Electrical Machinery)
  - Fabricated Metal Products and Housewares
- 7. 

  | Finished Apparel Products
- □ Paints, Varnishes and Industrial Chemicals (except Plastic Raw Materials)
  - 11. 

    Detroleum, Coal, Rubber, Stone and Glass Products
- ☐ Musical Instruments, Toys, Sporting Goods, Athletic Goods, Ordnance & Smokers' Supplies 12.
  - 13.
  - □ Jewelry and Fashion Accessories
     □ Furniture and Finished Wood Products 14.
    - 15.
- ☐ Leather and Leather Products ☐ MANUFACTURING, other than above. Please specify
- 17.  $\square$  Plastics Custom Molders, Extruders, Laminators, and Fabricators
- 18. 

  Plastic Materials
- ☐ Producers and Processors of Textiles, Lumber, Paper, Oils, Dyes, Chemicals, etc. used in Manufacture of 19.
- 20. 
   Plastic Machinery

## NON-MANUFACTURING

- 21.  $\Box$  Government: Federal, State, Municipal and Foreign; Officers of the Armed Forces
- 22. 

  Advertising Agencies, Sales Consultants and Sales Engineers
- 23. 

  Libraries, Schools, Colleges and Trade Associations
- 24.  $\square$  Consultants and Research Organizations, Architects, Engineers, Designers, Chemists
  - ☐ Transportation Operating Companies 26.
  - ☐ Retail Stores
- 27.  $\square$  Exporters, Importers, Distributors, Jobbers, Wholesalers and Manufacturers' Agents
- 28.  $\Box$  Doctors, Lawyers and other Professionals 29.  $\Box$  NON-MANUFACTURING, other than above. Please specify
- 30. Packaging & Containers
  - 31. ☐ Aerospace32. ☐ Construction Materials



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